AFWAL-TR-85-2049

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PROPERTIES OF AIRCRAFT FUELS AND RELATED MATERIALS

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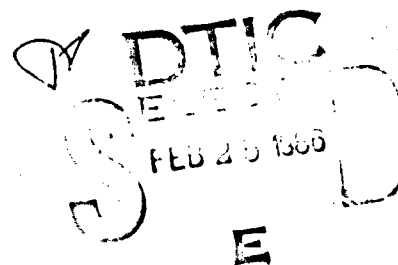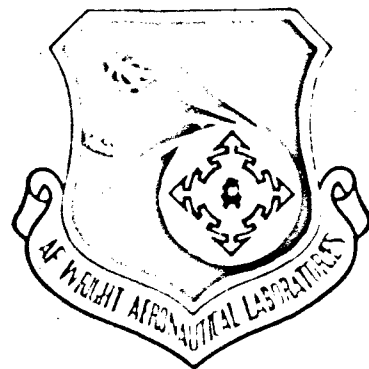DAYTON LABORATORY
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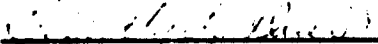WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-6563
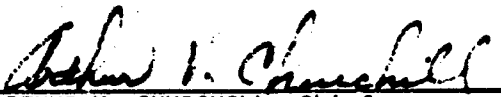
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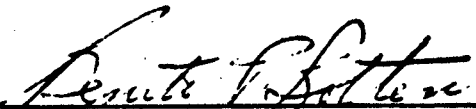
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This technical report has been reviewed and is approved for publication.

TIMOTHY L. DUES, Project Engineer
Fuels Branch

ARTHUR V. CHURCHILL, Chief
Fuels Branch
Fuels and Lubrication Division

FOR THE COMMANDER

BENITO P. BOTTERI, Assistant Chief
Fuels and Lubrication Division
Aero Propulsion Laboratory

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Fuel tests, analyses, and analytical method development were conducted on a number of fuels of an experimental nature in conjunction with ongoing Air Force programs for studying fuel combustion behavior, turbine engine design, and other fuel related technologies. Fuels from conventional and alternate sources were studied, as were fuels of the high density missile propellant type. A wide variety of both physical and chemical properties of the fuels were measured and are tabulated. Studies conducted to aid in the solution of operational problems are also reported. Keywords:

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FOREWORD

This final report was submitted by the Monsanto Company under Contract F33615-81-C-2035. This effort was sponsored by the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio under project No. 3048515 with Major Donald D. Potter serving as Contract Monitor during the first 18 months of the effort, and Mr. Tim L. Dues serving as Contract Monitor during the last 18 months of the effort. This report covers activities during the last 18 months of the contract; the first 18 months were covered by another report, AFWAL-TR-84-2022.

Much of the work performed during this phase of this program was planned in coordination with a number of other related fuel technology studies being conducted at, or under the sponsorship of, the Aero Propulsion Laboratory. Such efforts include research on turbine engine combustor design, fuel combustion behavior, improved fuel characterization methodology, and alternate fossil fuel source development. The experimental results presented in this report are intended to be used in the context of those research programs, and it is expected that the significance of the data will become apparent as the technical aspects of the related programs are published. In particular, many of the fuel specimens examined during this program were experimental in nature, and the various chemical and physical properties tabulated herein should not necessarily be regarded as characteristic of particular fuel types.

The authors wish to acknowledge the excellent guidance provided by Mr. Tim Dues during this phase of the work. Appreciation and acknowledgements are also expressed to members of the Monsanto Company staff: J. E. Strobel, B. M. Hughes, J. Giust, and M. Mason for their technical contributions, and to O. P. Tanner, Monsanto Company, St. Louis for the special data he has provided.

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SECTION I

INTRODUCTION AND SUMMARY

Many in-house and contractual programs covering all aspects of air-craft fuel technology are conducted by the Fuels Branch of the Fuels and Lubrication Division of the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories at Wright-Patterson Air Force Base. The basic objective of these programs is to ensure an adequate supply of dependable fuels for operational use by the Air Force. In order to achieve this goal, efforts are conducted in a number of broad areas of fuel technology: processing, availability, storage, transportation, distribution, handling, contamination composition, properties, quality, materials compatibility, combustion behavior, and environmental and health effects. Work performed under this contract has supported these efforts.

Chemical and physical characterization of jet fuels has been provided in support of the shale JP-4 pre-validation program, for studies correlating properties with engine test data, and for a number of other Air Force in-house and other contractual programs. Studies and analyses were conducted to determine the nature and source of fuel contaminants which had caused filter plugging; to determine level of trace metals which had caused thermal instability; to determine the interactive effects of fuel additives; and to aid in solving operational problems as they arose.

These studies and others that were required for this contractual effort are described in the sections that follow.

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SECTION II

FUELS CHARACTERIZATION

During the course of this program, a wide variety of conventional, experimental, alternate, and synthetic fuels have been characterized by chemical and physical means in order to correlate fuel characteristics with aircraft engine performance. The data presented in this section were obtained as a part of ongoing Air Force research programs either to aid in solving operational problems, or to define fuel composition and properties for specific engineering applications.

While not all programs required the same analytical measurements, data obtained on most samples included hydrocarbon type analysis (modified ASTM D 2789-71 and Monsanto Method 21-PQ-38-63), simulated distillation (ASTM D 2887-73), heat of combustion (ASTM D 240-76), true vapor pressure, kinematic viscosity (ASTM D 445-79), surface tension (capillary rise method), and density (dilatometer method).

1. DETAILED CHARACTERIZATION OF FUELS IN SUPPORT
    OF F100 TEST AND EVALUATION PROGRAM

Some 34 fuel samples were characterized in support of the F100 Program. A compilation of the samples, by number, follows.

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LIST OF FUELS

83-POSF-1006	83-POSF-1181	84-POSF-1723
83-POSF-1073	83-POSF-1253	84-POSF-1744
83-POSF-1074	83-POSF-1259	84-POSF-1951
83-POSF-1075	83-POSF-1268	84-POSF-1952
83-POSF-1076	83-POSF-1272	84-POSF-2034
83-POSF-1077	83-POSF-1273	84-POSF-2035
83-POSF-1084	83-POSF-1283	84-POSF-2036
83-POSF-1085	83-POSF-1284	84-POSF-2037
83-POSF-1086	83-POSF-1287	84-POSF-2038
83-POSF-1157	83-POSF-1297	84-POSF-2113
83-POSF-1158	83-POSF-1488	84-POSF-2114
83-POSF-1159		

All these fuels were JP-4 except for 84-POSF-1723 which was a
Jet A-1. Simulated distillation data for all these fuels are pre-
sented in Table 1; hydrocarbon type analyses in Table 2; heat of
combustion results in Table 3; and true vapor pressure, surface
tension, density, and kinematic viscosity data in Table 4. The
last four properties were determined as a function of temperature.

TABLE 1. SIMULATED DISTILLATION

% Recovered	83-POSF-1006 °C	83-POSF-1006 °F	83-POSF-1073 °C	83-POSF-1073 °F	83-POSF-1074 °C	83-POSF-1074 °F	83-POSF-1075 °C	83-POSF-1075 °F	83-POSF-1076 °C	83-POSF-1076 °F	83-POSF-1077 °C	83-POSF-1077 °F
0.5 (IBP)	29	84	30	86	29	83	27	81	29	84	29	84
1.0	31	87	32	90	35	95	28	83	35	95	34	94
5.0	58	137	59	139	56	134	59	138	57	135	56	133
10	78	172	73	163	66	150	87	189	67	152	62	144
20	99	209	91	196	89	193	117	243	95	203	87	189
30	112	233	102	216	112	233	126	259	117	243	111	232
40	127	261	118	244	124	256	136	277	139	282	142	288
50	141	286	135	275	142	288	141	286	163	326	172	341
60	152	305	150	303	163	326	145	293	187	369	189	373
70	164	327	168	335	186	367	164	328	206	402	206	402
80	175	347	191	376	209	408	196	385	224	435	223	433
90	195	383	221	43C	233	451	229	444	244	470	244	470
95	213	416	239	463	247	477	249	479	255	491	257	495
99	251	483	270	518	269	516	273	524	279	534	291	556
99.5 (FBP)	262	503	281	537	275	527	283	542	288	550	304	579

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TABLE 1 (continued)

% Recovered	83-POSF-1084 °C	°F	83-POSF-1085 °C	°F	83-POSF-1086 °C	°F	83-POSF-1157 °C	°F	83-POSF-1158 °C	°F	83-POSF-1159 °C	°F
0.5 (IBP)	27	80	34	93	27	80	27	81	27	80	27	81
1.0	28	82	37	98	28	82	32	90	28	83	30	85
5.0	58	137	66	150	59	138	35	95	65	149	57	135
10	77	170	79	175	74	166	60	141	83	181	71	160
20	98	209	91	196	97	207	96	205	101	213	98	208
30	116	241	100	212	114	238	118	244	121	250	124	255
40	136	277	109	228	127	261	141	286	139	282	149	301
50	159	317	116	241	142	287	160	320	159	317	170	338
60	180	356	124	255	160	319	175	346	177	350	185	366
70	202	396	135	274	181	358	192	378	198	388	200	392
80	222	432	152	306	209	409	208	406	220	428	216	421
90	245	472	215	419	238	460	228	443	244	471	235	456
95	260	501	249	479	258	496	244	471	260	500	252	485
99	305	581	289	552	293	559	276	529	288	550	285	545
99.5 (FBP)	318	604	303	577	305	580	288	550	296	565	295	563

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TABLE 1 (continued)

% Recovered	83-POSF-1181 °C	°F	83-POSF-1253 °C	°F	83-POSF-1259 °C	°F	83-POSF-1268 °C	°F	83-POSF-1272 °C	°F	83-POSF-1273 °C	°F
0.5 (IBP)	10	50	26	79	29	84	98	209	28	82	28	82
1.0	28	83	27	81	35	95	108	227	29	84	29	84
5.0	50	122	56	133	37	98	136	277	54	129	62	143
10	64	147	87	189	55	131	146	296	72	162	92	198
20	96	205	107	225	93	199	159	318	98	209	119	246
30	123	253	118	244	121	250	168	335	110	229	132	269
40	148	298	130	266	145	293	176	349	119	247	141	285
50	166	331	141	286	165	330	189	371	134	274	145	293
60	180	356	150	302	181	357	199	389	146	296	168	334
70	195	383	165	329	197	387	214	418	165	330	198	388
80	214	417	186	367	217	422	228	442	188	370	225	437
90	234	453	216	421	240	463	243	470	222	432	252	486
95	253	487	232	450	270	517	262	503	252	486	271	520
99	317	603	253	487	351	664	332	629	325	617	321	610
99.5 (FBP)	330	626	257	495	370	698	345	654	342	647	333	632

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TABLE 1 (continued)

% Recovered	83-POSF-1283 °C	83-POSF-1283 °F	83-POSF-1284 °C	83-POSF-1284 °F	83-POSF-1287 °C	83-POSF-1287 °F	83-POSF-1297 °C	83-POSF-1297 °F	83-POSF-1488 °C	83-POSF-1488 °F	84-POSF-1723 °C	84-POSF-1723 °F
0.5 (IBP)	29	84	31	88	28	82	29	85	27	80	130	265
1.0	34	94	33	92	34	93	31	87	28	83	140	283
5.0	66	151	60	140	58	137	67	152	58	136	157	315
10	82	179	73	163	73	163	84	183	72	161	164	325
20	100	212	97	207	100	213	99	211	99	210	175	346
30	127	260	110	230	126	258	108	226	119	246	185	364
40	152	305	124	255	149	300	118	245	139	282	196	384
50	173	344	139	282	167	332	126	259	157	315	207	405
60	189	371	154	310	183	361	139	282	175	347	219	426
70	205	401	173	343	199	391	151	305	192	378	232	449
80	226	439	200	391	217	423	180	355	210	410	247	476
90	252	486	246	475	238	450	220	427	230	446	263	506
95	282	539	283	541	259	498	248	478	242	468	276	529
99	351	663	322	611	313	595	293	559	266	510	303	578
99.5 (FBP)	373	702	334	634	330	626	311	591	275	526	310	590

TABLE 1 (continued)

% Recovered	84-POSF-1744 °C	84-POSF-1744 °F	84-POSF-1951 °C	84-POSF-1951 °F	84-POSF-1952 °C	84-POSF-1952 °F	84-POSF-2034 °C	84-POSF-2034 °F	84-POSF-2035 °C	84-POSF-2035 °F	84-POSF-2036 °C	84-POSF-2036 °F
0.5 (IBP)	28	82	27	81	31	88	29	84	118	244	29	84
1.0	30	85	29	84	32	90	30	86	126	259	34	93
5.0	58	136	54	129	58	136	36	96	150	302	54	129
10	74	165	65	149	70	158	57	135	162	324	66	150
20	102	215	95	203	100	212	90	193	175	346	88	191
30	122	251	127	261	133	271	110	230	187	368	109	229
40	136	277	153	307	161	322	130	266	195	383	135	276
50	150	302	171	340	177	351	151	304	204	400	158	317
60	166	330	185	365	192	378	171	339	215	419	174	346
70	184	362	196	385	203	397	187	369	223	434	192	378
80	205	401	213	415	219	426	204	399	235	455	209	409
90	230	446	231	448	236	457	223	434	251	484	231	447
95	245	473	244	471	248	478	235	456	264	507	244	471
99	267	513	270	518	269	516	256	493	284	543	264	507
99.5 (FBP)	278	532	284	543	275	527	263	505	288	550	271	520

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TABLE 1 (continued)

% Recovered	84-POSF-2037		84-POSF-2038	
	°C	°F	°C	°F
0.5 (IBP)	14	58	92	197
1.0	16	61	99	211
5.0	79	174	131	267
10	91	195	149	300
20	112	234	166	330
30	128	263	175	346
40	145	292	184	364
50	162	324	194	382
60	177	350	202	396
70	195	383	214	417
80	213	415	226	438
90	232	449	241	465
95	244	471	253	488
99	263	505	269	516
99.5 (FBP)	268	515	272	521

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TABLE 2. HYDROCARBON TYPE ANALYSES

Weight percent

	83-POSF-1006		83-POSF-1073		83-POSF-1074		83-POSF-1075		83-POSF-1076	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	51.4	43.1	42.8	37.1	60.2	54.1	66.0	59.2	56.0	48.0
Monocycloparaffins	30.5	-	37.9	-	22.3	-	13.7	-	21.5	-
Dicycloparaffins	2.9[d]	-	2.9[d]	-	4.2[d]	-	3.0[d]	-	5.7[d]	-
Total cycloparaffins	33.4	36.1	40.8	39.9	26.5[d]	28.1	16.7[d]	16.1	27.2[d]	27.6
Alkylbenzenes	14.3	20.8	14.6	22.1	10.5	15.9	15.8	23.9	12.9	19.8
Indans and tetralins	0.2	0	1.0	0.6	1.8	1.9	1.0	0.8	2.8	3.3
Indenes and dihydro-naphthalenes	-	0	-	0	-	u	-	0	-	0
Naphthalenes	0.7	0	0.8	0.3	1.0	0	0.5	0	1.1	1.3
Average carbon no.	8.2		8.3		8.5		8.9		9.1	

Weight percent

	83-POSF-1077		83-POSF-1084		83-POSF-1085		83-POSF-1086		83-POSF-1157	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	57.6	50.9	44.7	37.7	42.5	36.0	43.7	36.5	57.9	51.6
Monocycloparaffins	21.4	-	32.6	-	39.5	-	37.4	-	24.4	-
Dicycloparaffins	5.8[d]	-	4.4[d]	-	1.3[d]	-	5.4[d]	-	3.9[d]	-
Total cycloparaffins	27.2	27.9	37.0	37.2	40.8[d]	40.9	42.8[d]	45.4	28.3[d]	29.4
Alkylbenzenes	11.6	17.4	15.9	22.8	15.9	23.1	10.4	15.2	11.6	17.5
Indans and tetralins	2.4	2.7	1.5	1.4	0.3	0	2.2	2.5	1.5	1.4
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	1.2	1.1	0.9	0.9	0.5	0	0.9	0.4	0.7	0.1
Average carbon no.	8.9									

[a] Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b] Monsanto Method 21-PQ-38-63.

[c] Dash indicates method docs not provide information on these specific compound categories.

[d] Sum of two preceding values.

TABLE 2 (continued)

	Weight percent									
	83-POSF-1158		83-POSF-1159		83-POSF-1181		83-POSF-1253		83-POSF-1259	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	39.6	33.2	57.0	50.7	58.5	52.5	56.1	49.4	58.3	52.2
Monocycloparaffins	37.4	-	24.6	-	22.8	-	28.3	-	24.7	-
Dicycloparaffins	8.1[d]	-	4.4[d]	-	4.1[d]	-	3.2[d]	-	4.1[d]	-
Total cycloparaffins	45.5	46.3	29.0	30.1	26.9	27.4	31.5	33.1	28.8	30.1
Alkylbenzenes	11.0	11.6	11.2	16.9	12.3	18.6	11.2	16.7	10.7	16.2
Indans and tetralins	2.9	3.5	1.9	2.0	1.6	1.5	0.9	0.8	1.5	1.5
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	1.0	1.0	0.8	0.3	0.7	0	0.3	0	0.7	0
Averae carbon no.			8.1		8.7		8.7		8.7	

	Weight percent									
	83-POSF-1268		83-POSF-1272		84-POSF-1273		84-POSF-1283		84-POSF-1284	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	50.1	47.8	50.7	43.1	64.6	57.7	54.3	49.1	49.5	41.5
Monocycloparaffins	34.7	-	30.6	-	16.3	-	25.4	-	31.0	-
Dicycloparaffins	0	-	2.6[d]	-	4.1[d]	-	5.4[d]	-	2.3[d]	-
Total cycloparaffins	34.7[d]	33.1	33.2	35.0	20.4	20.6	30.8	30.2	33.3	34.5
Alkylbenzenes	12.3	16.2	14.5	21.8	12.7	19.6	12.1	17.9	15.7	24.0
Indans and tetralins	1.5	1.6	0.8	0.1	1.5	1.6	2.0	2.1	0.7	0
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	1.4	1.3	0.8	0	0.8	0.5	0.8	0.7	0.8	0
Average carbon no.	10.1		8.1		9.1		9.1		8.2	

[a] Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b] Monsanto Method 21-P2-38-63.

[c] Dash indicates method does not provide information on these specific compound categories.

[d] Sum of two preceding values.

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TABLE 2 (continued)

	Weight percent									
	83-POSF-1287		83-POSF-1297		83-POSF-1488		84-POSF-1723		84-POSF-1744	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	57.5	51.9	55.2	49.4	62.4	56.7	38.3	37.6	63.4	57.8
Monocycloparaffins	26.2	-	27.4	-	20.9	-	41.1	-	19.0	-
Dicycloparaffins	4.3	-	2.2	-	2.5	-	0.5	-	2.1	-
Total cycloparaffins	30.5	31.4	29.6	29.6	23.4[d]	23.3	41.6[d]	40.6	21.1	21.0
Alkylbenzenes	9.8	14.8	13.7	20.9	12.1	18.4	12.7	15.0	13.4	20.5
Indans and tetralins	1.6	1.7	0.6	0.1	1.6	1.6	4.4	3.8	1.1	0.7
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	0.6	0.2	0.9	0	0.5	0	3.0	3.0	1.0	0
Average carbon no.	8.9		8.3		8.7		10.6		8.5	

	Weight percent									
	84-POSF-1951		83-POSF-1952		84-POSF-2034		84-POSF-2035		84-POSF-2036	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	64.6	59.4	64.6	59.5	53.3	45.9	48.8	48.1	58.5	52.5
Monocycloparaffins	16.9	-	17.3	-	27.9	-	34.8	-	24.8	-
Dicycloparaffins	2.6[d]	-	2.5	-	4.9	-	0	-	4.0	-
Total cycloparaffins	19.5	19.5	19.8[d]	19.4	38.8[d]	35.3	34.8[d]	33.9	28.8[d]	30.0
Alkylbenzenes	13.3	19.7	13.1	19.6	11.1	16.7	10.6	13.2	10.3	15.8
Indans and tetralins	1.6	1.4	1.7	1.5	1.8	1.8	4.7	3.9	1.5	1.5
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	0.8	0	0.8	0	1.0	0.3	1.1	0.9	0.9	0.2
Average carbon no.	8.6		8.7		8.6		10.5		8.7	

(continued)

TABLE 2 (continued

| | Weight percent | | | |
| | 84-POSF-2037 | | 84-POSF-2038 | |
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	56.1	51.0	43.7	41.9
Monocycloparaffins	27.5	-	36.9	-
Dicycloparaffins	5.3[d]	-	0	-
Total cycloparaffins	32.8	33.6	36.9[d]	36.1
Alkylbenzenes	9.5	13.7	13.6	16.8
Indans and tetralins	1.5	1.7	3.1	2.7
Indenes and dihydro-naphthalenes	-	0	-	0
Naphthalenes	0.1	0	2.7	2.5
Average carbon no.	9.0		10.1	

[a]Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b]Monsanto Method 21-PQ-38-63.

[c]Dash indicates method does not provide information on these specific compound categories.

[d]Sum of two preceding values.

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TABLE 3. HEAT OF COMBUSTION DATA

Sample number	Gross, BTU/lb			Net, BTU/lb[a]
	1	2	Avg.	
83-POSF-1006	20,041	20,038	20,040	20,040 - (91.23x%H)
83-POSF-1073	19,941	19,900	19,920	19,920 - (91.23x%H)
83-POSF-1074	20,008	20,024	20,016	20,016 - (91.23x%H)
83-POSF-1075	20,028	20,042	20,035	20,035 - (91.23x%H)
83-POSF-1076	20,013	19,984	19,998	19,998 - (91.23x%H)
83-POSF-1077	19,984	20,014	19,999	19,999 - (91.23x%H)
83-POSF-1084	19,858	19,869	19,864	19,864 - (91.23x%H)
83-POSF-1085	19,962	19,992	19,977	19,977 - (91.23x%H)
83-POSF-1086	19,977	19,950	19,964	19,964 - (91.23x%H)
83-POSF-1157	19,984	20,020	20,002	20,002 - (91.23x%H)
83-POSF-1158	19,831	19,860	19,846	19,846 - (91.23x%H)
83-POSF-1181	19,961	19,942	19,952	19,952 - (91.23x%H)
83-POSF-1253	20,030	20,032	20,031	20,031 - (91.23x%H)
83-POSF-1259	20,045	20,008	20,027	20,027 - (91.23x%H)
83-POSF-1268	19,888	19,863	19,876	19,876 - (91.23x%H)
83-POSF-1272	19,952	19,960	19,956	19,956 - (91.23x%H)
83-POSF-1273	20,014	20,032	20,023	20,023 - (91.23x%H)
83-POSF-1283	20,017	20,034	20,026	20,026 - (91.23x%H)
83-POSF-1284	19,886	19,894	19,390	19,890 - (91.23x%H)
83-POSF-1287	20,020	20,058	20,039	20,039 - (91.23x%H)
83-POSF-1297	20,023	20,006	20,014	20,014 - (91.23x%H)
84-POSF-1723	19,899	19,952	19,926	19,926 - (91.23x%H)
84-POSF-1744	20,371	20,356	20,363	20,363 - (91.23x%H)
83-POSF-1488	20,045	20,052	20,049	20,049 - (91.23x%H)
84-POSF-1951	19,948	19,950	19,949	19,949 - (91.23x%H)
84-POSF-1952	20,003	19,958	19,980	19,980 - (91.23x%H)
84-POSF-2034	19,925	19,921	19,923	19,923 - (91.23x%H)
84-POSF-2035	19,946	19,882	19,914	19,914 - (91.23x%H)
84-POSF-2036	19,995	20,045	20,020	20,020 - (91.23x%H)
84-POSF-2037	20,038	20,023	20,031	20,031 - (91.23x%H)
84-POSF-2038	19,795	19,820	19,808	19,908 - (91.23x%H)

[a]The value for hydrogen content of the sample (% H) is not available at this time. The net heat of combustion can be calculated using this equation when % H is obtained.

TABLE 4. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1006				
-30.1°F	-	2.013	-	-
-20°F	8[a]	1.785	0.7936	28.1[b]
-4°F	-	1.512	-	-
32°F	33	1.139	0.7718	25.4
59°F	-	-	0.7589	-
70°F	74	0.863	0.7546	23.4
100°F	133	0.720	0.7409	21.9
140°F	270	0.583	0.7230	19.8
83-POSF-1073				
-30.1°F	-	2.010	-	-
-20°F	8[a]	1.798	0.7998	28.2[b]
-4°F	-	1.514	-	-
32°F	32	1.135	0.7767	25.4
59°F	-	-	0.7653	-
70°F	76	0.862	0.7608	23.4
100°F	134	0.719	0.7471	21.8
140°F	290	0.582	0.7296	19.7
83-POSF-1074				
-30.1°F	-	2.120	-	-
-20°F	7[a]	1.876	0.7899	27.4[b]
-4°F	-	1.580	-	-
32°F	32	1.174	0.7678	24.7
59°F	-	-	0.7557	-
70°F	74	0.887	0.7512	22.8
100°F	138	0.740	0.7377	21.2
140°F	285	0.596	0.7199	19.2
83-POSF-1075				
-30.1°F	-	2.048	-	-
-20°F	7[a]	1.818	0.7895	27.8[b]
-4°F	-	1.521	-	-
32°F	30	1.149	0.7681	25.1
59°F	-	-	0.7563	-
70°F	70	0.872	0.7513	23.2
100°F	125	0.719	0.7378	21.6
140°F	255	0.587	0.7206	19.6

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm³)	Surface tension (dynes/cm)
83-POSF-1076				
-30.1°F	-	2.464	-	-
-20°F	7[a]	2.185	0.8014	28.1[b]
-4°F	-	1.810	-	-
32°F	30	1.334	0.7806	25.4
59°F	-	-	0.7690	-
70°F	71	0.981	0.7643	23.5
100°F	130	0.806	0.7508	21.8
140°F	268	0.647	0.7327	19.8
83-POSF-1077				
-30.1°F	-	2.404	-	-
-20°F	6[a]	2.116	0.7971	27.7[b]
-4°F	-	1.761	-	-
32°F	29	1.284	0.7745	25.1
59°F	-	-	0.7642	-
70°F	71	0.969	0.7592	23.2
100°F	137	0.797	0.7457	21.7
140°F	282	0.639	0.7293	19.6
83-POSF-1084				
-30.1°F	-	2.345	-	-
-20°F	7[a]	2.072	0.8051	28.4[b]
-4°F	-	1.755	-	-
32°F	32	1.283	0.7850	25.9
59°F	-	-	0.7738	-
70°F	78	0.9660	0.7688	24.0
100°F	145	0.7694	0.7554	22.6
140°F	305	0.6359	0.7369	20.6
83-POSF-1085				
-30.1°F	-	1.629	-	-
-20°F	7[a]	1.471	0.7928	27.8[b]
-4°F	-	1.277	-	-
32°F	32	0.9676	0.7710	25.1
59°F	-	-	0.7583	-
70°F	76	0.7587	0.7539	23.2
100°F	140	0.6375	0.7398	21.2
140°F	288	0.5201	0.7214	19.5

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1086				
-30.1°F	-[a]	2.188	-	-[b]
-20°F	6	1.942	0.8023	28.3
-4°F	-	1.653	-	-
32°F	26	1.224	0.7615	25.6
59°F	-	-	0.7695	-
70°F	66	0.9254	0.7643	23.6
100°F	125	0.7620	0.7507	22.0
140°F	270	0.6153	0.7326	19.8
83-POSF-1157				
-30.1°F	-[a]	2.373	-	-[b]
-20°F	6	2.088	0.7939	28.9
-4°F	-	1.796	-	-
32°F	28	1.295	0.7722	26.2
59°F	-	-	0.7614	-
70°F	68	0.972	0.7565	24.3
100°F	128	0.799	0.7431	22.6
140°F	265	0.644	0.7259	20.4
83-POSF-1158				
-30.1°F	-[a]	2.603	-	-[b]
-20°F	7	2.262	0.8143	29.2
-4°F	-	1.917	-	-
32°F	30	1.391	0.7936	26.6
59°F	-	-	0.7810	-
70°F	70	1.028	0.7761	24.8
100°F	128	0.8430	0.7627	23.3
140°F	265	0.6703	0.7444	21.4
83-POSF-1159				
-30.1°F	-[a]	2.372	-	-[b]
-20°F	8	2.084	0.7966	28.2
-4°F	-	1.775	-	-
32°F	37	1.304	0.7726	25.8
59°F	-	-	0.7627	-
70°F	78	0.9729	0.7577	24.0
100°F	140	0.7998	0.7444	22.6
140°F	290	0.6423	0.7276	20.8

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1181				
-30.1°F	-	2.325	-	-[b]
-20°F	8.1[a]	2.054	0.7955	28.8[b]
-4°F	-	1.731	-	-
32°F	33	1.282	0.7723	26.1
59°F	-	-	0.7617	-
70°F	76	0.958	0.7568	24.0
100°F	138	0.791	0.7434	22.4
140°F	278	0.640	0.7264	20.4
83-POSF-1253				
-30.1°F	-[a]	2.128	-	-[b]
-20°F	10[a]	1.818	0.7904	28.4[b]
-4°F	-	1.549	-	-
32°F	39	1.163	0.7672	25.7
59°F	-	-	0.7562	-
70°F	85	0.879	0.7512	23.8
100°F	148	0.733	0.7378	22.1
140°F	284	0.594	0.7202	20.2
83-POSF-1259				
-30.1°F	-	2.514	-	-[b]
-20°F	6.4[a]	2.225	0.7950	28.3[b]
-4°F	-	1.825	-	-
32°F	29	1.334	0.7732	25.5
59°F	-	-	0.7619	-
70°F	71	0.999	0.7571	23.5
100°F	135	0.829	0.7438	21.8
140°F	288	0.660	0.7272	19.7
83-POSF-1268				
-30.1°F	-	4.425	-	-[b]
-20°F	0.7[a]	3.775	0.8200	29.9[b]
-4°F	-	2.937	-	-
32°F	3.3	1.996	0.7993	27.4
59°F	-	-	0.7885	-
70°F	8.2	1.395	0.7839	25.8
100°F	15.5	1.129	0.7713	24.3
140°F	34	0.853	0.7546	22.4

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm³)	Surface tension (dynes/cm)
83-POSF-1272				
-30.1°F	-	1.908	-	-
-20°F	6.1[a]	1.687	0.7940	29.3[b]
-4°F	-	1.440	-	-
32°F	28	1.091	0.7722	26.0
59°F	-	-	0.7599	-
70°F	69	0.850	0.7550	23.6
100°F	130	0.701	0.7412	21.7
140°F	276	0.569	0.7238	19.2
83-POSF-1273				
-30.1°F	-	2.242	-	-
-20°F	8.9[a]	2.061	0.7944	27.6[b]
-4°F	-	1.703	-	-
32°F	35	1.256	0.7730	25.2
59°F	-	-	0.7606	-
70°F	77	0.941	0.7588	23.2
100°F	133	0.776	0.7424	21.9
140°F	265	0.630	0.7256	20.0
83-POSF-1283				
-30.1°F	-	2.628	-	-
-20°F	4.9[a]	2.326	0.8019	27.9[b]
-4°F	-	1.901	-	-
32°F	23.5	1.382	0.7812	25.5
59°F	-	-	0.7688	-
70°F	59	1.024	0.7639	23.8
100°F	114	0.848	0.7507	22.2
140°F	245	0.664	0.7336	20.5
83-POSF-1284				
-30.1°F	-	1.868	-	-
-20°F	6.8[a]	1.702	0.7956	28.4[b]
-4°F	-	1.429	-	-
32°F	32	1.085	0.7746	25.5
59°F	-	-	0.7616	-
70°F	75	0.834	0.7566	23.3
100°F	140	0.695	0.7429	21.5
140°F	300	0.578	0.7253	19.4

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1287				
-30.1°F	-	2.516	-	-
-20°F	5.2[a]	2.238	0.7946	28.4[b]
-4°F	-	1.839	-	-
32°F	25	1.357	0.7742	25.7
59°F	-	-	0.7617	-
70°F	65	1.005	0.7569	23.6
100°F	124	0.829	0.7437	22.1
140°F	270	0.662	0.7265	20.0
83-POSF-1297				
-30.1°F	-	1.809	-	-
-20°F	6.6	1.636	0.7886	28.2[b]
-4°F	-	1.389	-	-
32°F	32	1.062	0.7675	25.4
59°F	-	-	0.7552	-
70°F	76	0.814	0.7502	23.4
100°F	135	0.681	0.7369	21.8
140°F	276	0.576	0.7190	19.6
83-POSF-1488				
-30.1°F	-	2.2620	-	-
-20°F	9[a]	2.0192	0.7892	28.3[b]
-4°F	-	1.6958	-	-
32°F	34	1.2640	0.7690	25.8
59°F	-	-	0.7577	-
70°F	76	0.9499	0.7530	24.0
100°F	132	0.7837	0.7395	22.6
140°F	262	0.6303	0.7217	20.6
84-POSF-1723				
-30.1°F	-	7.6082	-	-
-20°F	5.8[a]	6.1395	0.8432	32.5[b]
32°F	12	2.8664	0.8229	29.7
59°F	-	-	0.8132	-
70°F	18	1.8852	0.8080	27.6
100°F	25	1.4388	0.7963	25.9
140°F	36	1.0752	0.7799	23.7

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
84-POSF-1741				
-30.1°F	-	7.6082	-	-[a]
-20°F	8.4	1.9598	0.7893	28.1[a]
32°F	34	1.2327	0.7672	25.6
59°F	-	-	0.7559	-
70°F	76	0.9280	0.7510	23.7
100°F	136	0.7701	0.7382	22.3
140°F	269	0.6191	0.7212	20.3
84-POSF-1951				
-30.1°F	-	2.4557	-	-[b]
-20°F	8.3[a]	2.1669	0.7920	28.9[b]
-4°F	-	1.8133	-	-
32°F	35.5	1.3327	0.7696	26.1
59°F	-	-	0.7580	-
70°F	80	0.9968	0.7533	24.0
100°F	144	0.8234	0.7404	22.4
140°F	296	0.6587	0.7232	20.2
84-POSF-1952				
-30.1°F	-	2.4995	-	-[b]
-20°F	7.4[a]	2.2111	0.7901	28.9[b]
-4°F	-	1.8464	-	-
32°F	38	1.3503	0.7689	26.2
59°F	-	-	0.7579	-
70°	82	1.0113	0.7534	24.2
100°F	155	0.8325	0.7412	22.6
140°F	335	0.6685	0.7249	20.5
84-POSF-2034				
-30.1°F	-	2.3460	-	-[b]
-20°F	8.2[a]	2.1091	0.7993	29.0[b]
-4°F	-	1.7714	-	-
32°F	34	1.3080	0.7771	26.3
59°F	-	-	0.7656	-
70°F	78	0.9699	0.7609	24.3
100°F	138	0.8060	0.7481	22.7
140°F	277	0.6461	0.7310	20.6

TABLE 4 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
84-POSF-2035				
-30.1°F	-	6.4397	-	-
-20°F	4.4[a]	5.4077	0.8295	31.6[b]
-4°F	-	4.1337	-	-
32°F	9	2.6412	0.8098	28.8
59°F	-	-	0.7984	-
70°F	15	1.7432	0.7934	26.7
100°F	21.5	1.3587	0.7825	25.1
140°F	33.5	1.0246	0.7660	22.9
84-POSF-2036				
-30°F	-	2.3508	-	-
-20°F	10.3[a]	2.0976	0.7940	28.6[b]
-4°F	-	1.7578	-	-
32°F	32.5	1.2938	0.7720	25.9
59°F	-	-	0.7606	-
70°F	75	0.9678	0.7560	23.9
100°F	137	0.0854	0.7443	22.3
140°F	275	0.6466	0.7264	20.2
84-POSF-2037				
-30.1°F	-	2.7255	-	-
-20°F	15[a]	2.4135	0.8009	28.9[b]
-4°F	-	1.9835	-	-
32°F	47.5	1.4392	0.7789	26.2
59°F	-	-	0.7675	-
70°F	95	1.0577	0.7628	24.3
100°F	152	0.8709	0.7502	22.8
140°F	272	0.6953	0.7333	20.8
84-POSF-2038				
-30.1°F	-	5.1930	-	-
-20°F	10[a]	4.4094	0.8335	31.3[b]
-4°F	-	3.4389	-	-
32°F	20.5	2.2706	0.8125	28.7
59°F	-	-	0.8016	-
70°F	31.5	1.5478	0.7971	26.8
100°F	42.5	1.2228	0.7850	25.3
140°F	61	0.9307	0.7689	23.3

[a]Value determined by extrapolation of log P versus 1/T vapor pressure relationship.

[b]Value obtained by linear regression extrapolation of data.

Thermal conductivity at three different temperatures was determined for some of the F100 fuels using the transient hot wire method (1). This work was conducted at Monsanto's Central Research Laboratories in St. Louis. Each fuel was analyzed 8-10 times, i.e., at four or five power levels and two different time increments. Data are presented in Table 5 along with values for relative standard deviation of the individual analysis results from the average. Plots of thermal conductivity versus temperature are presented in Figures 1 and 2. Constants for a polynomial equation which can be used for calculation of thermal conductivity at other temperatures are given in Table 6. These data were used to calculate thermal conductivities at 0°, 20° and 40°C as shown in Table 5.

Specific heat analyses on some fuels, reported in Table 7, were carried out at Monsanto, St. Louis by differential scanning calorimetry (DSC). The instrumentation used was a Perkin-Elmer DSC-II C with an intracooler II subambient accessory and a Thermal Analysis Data Station computer system for data acquisition and manipulation.

TABLE 5. THERMAL CONDUCTIVITY OF FUELS BY THE TRANSIENT HOT WIRE METHOD

Sample no.	Average values[a] and precision						Values calculated from curve fitting data, Table 6		
	0°C		20°C		40°C		0°C	20°C	40°C
	W/m·°K[b]	% SD[c]	W/m·°K[b]	% SD[c]	W/m·°K[b]	% SD[c]			
83-POSF-1006	0.123	±2.12%	0.117	±2.69%	0.110	±2.92%	0.123	0.117	0.110
83-POSF-1073	0.123	±4.05%	0.116	±2.40%	0.113	±2.49%	0.122	0.117	0.112
83-POSF-1074	0.123	±1.26%	0.116	±1.34%	0.114	±2.55%	0.123	0.118	0.113
83-POSF-1075	0.123	±1.50%	0.118	±1.88%	0.114	±1.71%	0.123	0.118	0.114
83-POSF-1076	0.122	±3.11%	0.119	±3.65%	0.111	±3.05%	0.122	0.117	0.112
83-POSF-1077	0.122	±1.62%	0.117	±1.62%	0.113	±1.80%	0.121	0.117	0.113
Toluene standard	0.140	±3.71%	0.132	±2.12%	0.125	±3.05%	0.140	0.132	0.125
Toluene lit values	0.1366		0.1308		0.1250				

[a] Average of 8-10 measurements (4-5 current settings, each over two different time intervals.)

[b] Thermal conductivity, SI unit of measurement, watts per meter per degree kelvin (equals 1.7307 Btu/ft·hr·°F).

[c] Percent relative standard deviation of individual measurements from the average.

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TABLE 6.   CONSTANTS FROM CURVE FITTING DATA FROM SAMPLE(S)

CONSTANTS FROM CURVE FITTING DATA FROM SAMPLE(S)

$Y = A0 + A1*X + A2*X*X$

Y : THERMAL CONDUCTIVITY (W/m-K)
X : TEMPERATURE (DEG C)

CC = CORRELATION COEFFICIENT

SAMPLE NAME	A0	A1	A2	CC	95% LIMITS
JET FUEL 03-POBF-1066	.12929	-.003130	.000000000	-.876	.00576
JET FUEL, 03-POBF-1073	.122031	-.002494	.000000000	-.747	.00746
JET FUEL: 03-POBF-1074	.122007	-.002455	.000000000	-.003	.00442
JET FUEL: 03-POBF-1075	.123011	-.002360	.000000000	-.098	.00395
JET FUEL: 03-POBF-1076	.122414	-.008502	.000000000	-.749	.00762
JET FUEL: 03-POBF-1077	.121295	-.002163	.000000000	-.005	.00390
TOLUENE STANDARD	.139593	-.003661	.000000000	-.034	.00760

=== END OF DATA ===
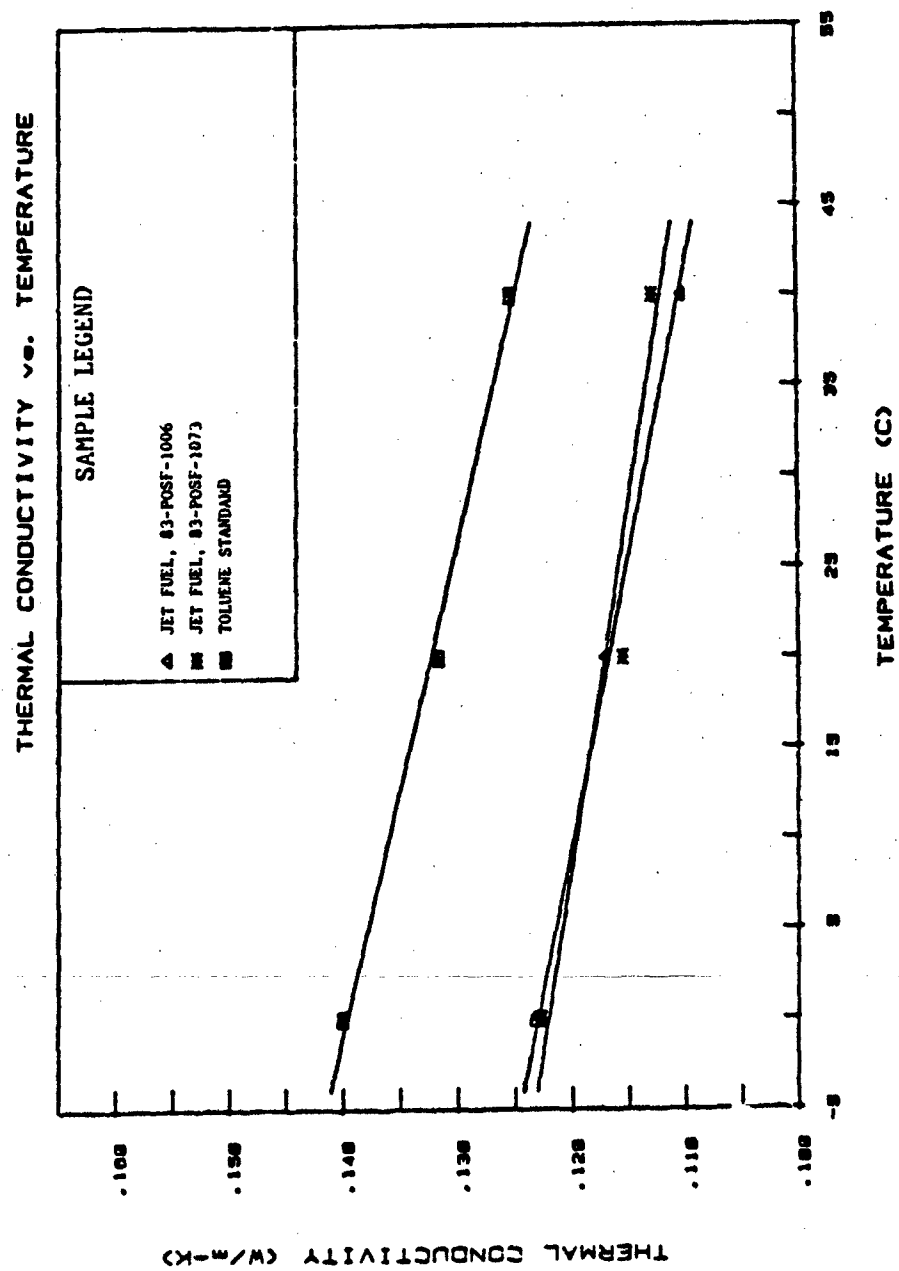
Figure 1. Thermal conductivity versus temperature.
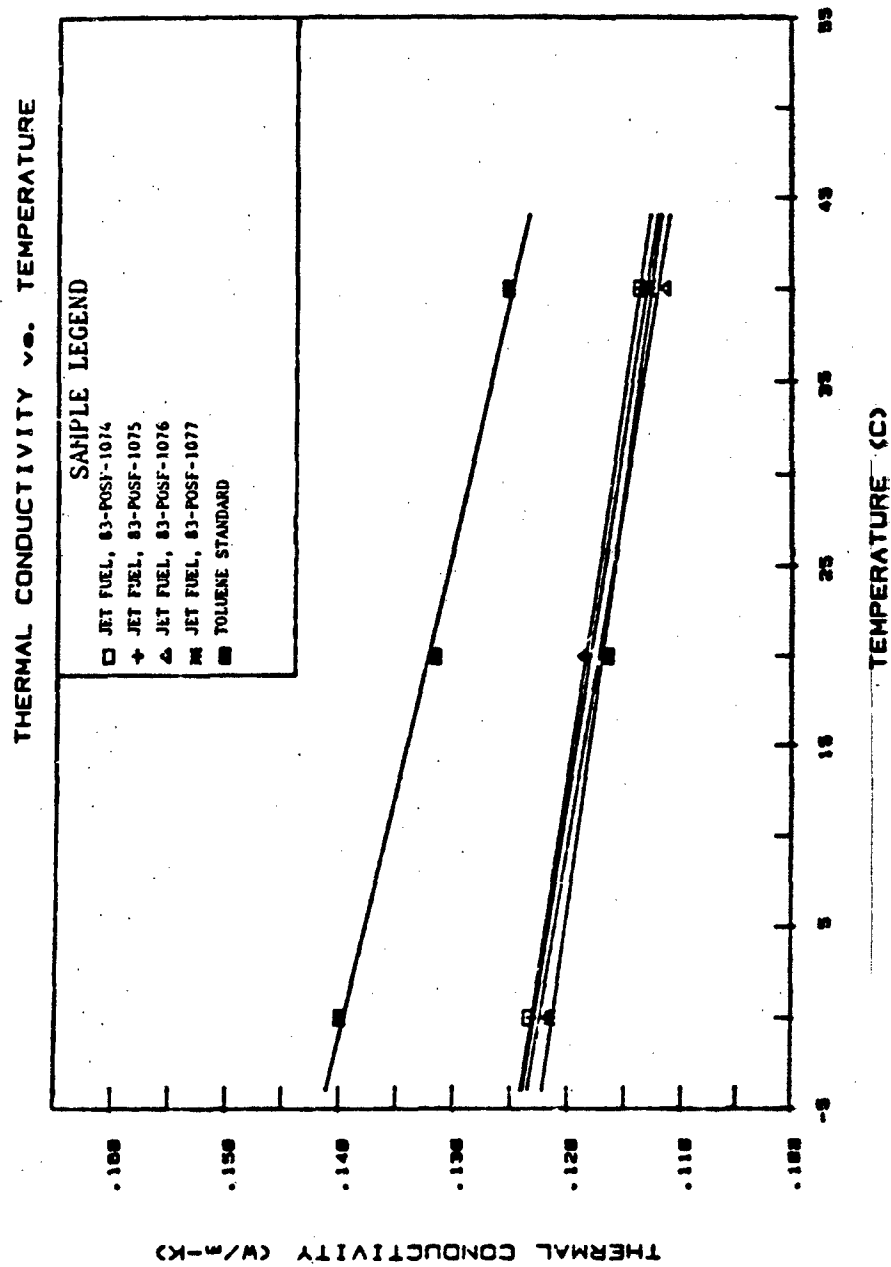
Figure 2. Thermal conductivity versus temperature.

TABLE 7. SPECIFIC HEAT ANALYSES

Sample number JP-4	Cal/g°C or Btu/lb°F		
	4°F(-15.6°C)	60°F(15.6°C)	104°F(37.8°C)
84-POSF-1723	0.433 ± .004	0.457 ± .004	0.477 ± .005
84-POSF-1744	0.449 ± .002	0.469 ± .002	0.490 ± .002
84-POSF-1951	0.445 ± 0.002	0.478 ± 0.001	0.500 ± 0.002
84-POSF-1952	0.463 ± 0.002	0.496 ± 0.003	0.521 ± 0.002
84-POSF-2034	0.442 ± 0.002	0.473 ± 0.000	0.495 ± 0.004
84-POSF-2035	0.416 ± 0.004	0.450 ± 0.001	0.475 ± 0.000
84-POSF-2036	0.429 ± 0.003	0.467 ± 0.003	0.485 ± 0.002
84-POSF-2037	0.423 ± 0.003	0.451 ± 0.001	0.479 ± 0.002
Heptane (>99% purity)	0.499 ± 0.000	0.530 ± 0.000	0.558 ± 0.001
Heptane (Literature value)[a]	0.505	0.528	0.550

[a]API project 44, Table 23-2-(1.202)-VC

2. CHARACTERIZATION OF TURBINE ENGINE FUEL THERMAL COKING PROGRAM

Twelve turbine engine fuels were characterized in detail by means of simulated distillation (ASTM D 2887), hydrocarbon type analysis, elemental analysis (carbon, hydrogen, oxygen and nitrogen), and heat of combustion (ASTM D 240-76). Results of these analyses are compiled in Tables 8-11.

Identificaton of the fuels as to sample number and type of fuel is as follows:

Sample No.	Type	Sample No.	Type
82-POSF-0167	JP-7	83-POSF-0969	JP-4
82-POSF-0168	JP-7	83-POSF-0970	JP-7
82-POSF-0414	JP-4	83-POSF-0971	JP-8
82-POSF-0417	JP-7	83-POSF-0972	JP-4
82-POSF-0418	JP-8	83-POSF-0973	JP-4
82-POSF-0477	JP-4		
82-POSF-0968	Occidental Light Distillate		

TABLE 8. SIMULATED DISTILLATION

% Recovered	82-POSF-0167		82-POSF-0168		82-POSF-0414		82-POSF-0417		82-POSF-0418		82-POSF-0477	
	°C	°F	°C	°F	°C	°F	°C	°F	°C	°F	°C	°F
0.5 (IBP)	147	297	34	92	35	95	35	95	35	95	35	95
1.0	152	305	35	95	53	128	36	96	36	96	52	126
5.0	173	343	36	97	82	179	166	331	135	276	81	178
10	185	365	131	269	90	194	181	358	152	305	89	193
20	198	389	162	323	107	224	197	387	168	335	106	222
30	210	410	174	346	119	246	209	408	179	354	118	245
40	218	424	184	363	140	284	217	422	188	371	138	280
50	224	435	194	382	162	323	224	434	197	387	158	317
60	231	448	203	397	182	359	231	448	207	404	179	354
70	237	459	214	417	203	397	237	458	217	422	200	392
80	246	474	226	438	224	435	246	475	228	442	221	430
90	254	489	243	469	250	482	255	490	244	472	246	475
95	261	502	264	506	269	517	264	507	264	506	264	506
99	278	532	307	584	312	594	290	553	322	612	317	602
99.5 (FBP)	289	552	316	601	320	608	300	572	335	635	332	629

TABLE 8. (continued)

% Recovered	83-POSF-0968		83-POSF-0969		83-POSF-0970		83-POSF-0971		83-POSF-0972		83-POSF-0973	
	°C	°F	°C	°F	°C	°F	°C	°F	°C	°F	°C	°F
0.5 (IBP)	115	239	35	95	37	98	113	236	36	97	34	93
1.0	126	259	37	98	143	289	121	250	39	103	35	95
5.0	160	319	86	188	172	341	146	295	87	189	72	161
10	173	343	92	197	186	366	158	317	97	206	89	192
20	189	372	116	242	200	392	171	340	118	245	101	214
30	201	393	135	276	211	412	180	356	143	289	118	245
40	214	417	163	326	219	426	189	372	173	344	134	273
50	221	430	188	370	226	439	197	387	196	384	152	306
60	231	448	207	404	233	451	207	404	212	414	174	346
70	240	464	227	441	239	462	216	421	229	443	195	384
80	254	490	236	457	248	479	227	440	234	454	217	422
90	285	545	257	494	258	497	241	466	253	488	240	464
95	342	647	278	532	267	513	257	495	270	518	256	492
99	421	789	326	618	324	616	313	595	307	585	296	565
99.5 (FBP)	430	805	336	637	336	637	327	621	323	613	317	603

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TABLE 9. HYDROCARBON TYPE ANALYSES

Weight percent

	82-POSF-0167		82-POSF-0168		82-POSF-0414		82-POSF-0417		82-POSF-0418	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	48.2	48.5	46.7	45.7	61.9	57.9	48.0	48.1	46.9	45.9
Monocycloparaffins	45.4	-	35.1	-	23.5	-	45.5	-	35.0	-
Dicycloparaffins	2.4[d]	-	0	-	5.5	-	2.8[d]	-	0	-
Total cycloparaffins	47.8[d]	47.3	35.1[d]	33.6	29.0[d]	29.5	48.3	48.0	35.0[d]	33.5
Alkylbenzenes	2.9	2.8	13.4	16.8	7.6	11.4	2.9	2.7	13.4	16.8
Indans and tetralins	0.8	0.8	4.2	3.5	1.1	1.2	0.5	0.6	4.2	3.5
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	0.3	0.6	0.6	0.4	0.4	0	0.3	0.6	0.5	0.3
Average carbon no.	12.1		10.1		9.0		12.2		10.1	

Weight percent

	82-POSF-0477		83-POSF-0968		83-POSF-0969		83-POSF-0970		83-POSF-0971	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	61.9	58.1	36.4	35.2	45.1	44.0	48.3	48.5	46.9	45.9
Monocycloparaffins	23.5	-	36.2	-	27.2	-	46.4	-	34.9	-
Dicycloparaffins	5.4[d]	-	1.9	-	0	-	2.0[d]	-	0	-
Total cycloparaffins	28.9[d]	29.3	38.1[d]	36.8	27.2[d]	22.3	48.4[d]	48.0	34.9[d]	33.5
Alkylbenzenes	7.6	11.4	14.2	16.8	10.1	14.0	2.8	2.5	13.4	16.7
Indans and tetralins	1.1	1.2	7.9	6.7	2.9	2.6	0.4	0.6	4.2	3.5
Indenes and dihydro-naphthalenes	-	0	-	1.5	-	0.3	-	0	-	0
Naphthalenes	0.5	0	3.4	3.0	14.7	16.8	0.1	0.4	0.6	0.4
Average carbon no.	9.0		10.1		10.0		12.1		10.0	

TABLE 9. (continued)

	Weight percent			
	83-POSF-0972		83-POSF-0973	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	38.2	36.1	61.1	57.8
Monocyclcparaffins	23.2	-	23.8	-
Dicycloparaffins	0	-	5.5[d]	-
Total cycloparaffins	23.2[d]	18.2	29.3[d]	29.6
Alkylbenzenes	11.3	15.6	7.6	11.4
Indans and tetralins	4.0	3.5	1.1	1.2
Indenes and dihydro- naphthalenes	-	0.5	-	0
Naphthalenes	23.3	26.1	0.4	0
Average carbon no.	10.1		9.0	

[a]Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b]Monsanto Method 21-PQ-38-63.

[c]Dash indicates method does not provide information on these specific compound categories.

[d]Sum of two preceding values.

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TABLE 10. ELEMENTAL ANALYSES

	Weight Percent			
	Carbon	Hydrogen	Nitrogen	Oxygen
82-POSF-0167	85.45	14.15	.0051	.005
82-POSF-0168	86.58	13.89	<.0006	.038
82-POSF-0414	86.48	13.90	<.0006	.015
82-POSF-0417	85.39	14.65	.0013	≤.005
82-POSF-0418	86.73	13.94	<.0006	.006
82-POSF-0477	86.49	13.79	<.0006	.067
83-POSF-0968	86.02	13.58	.64	.625
83-POSF-0969	87.88	12.82	.0023	.017
83-POSF-0970	86.07	14.39	.0022	.008
83-POSF-0971	86.25	14.05	.0014	≤.006
83-POSF-0972	87.57	12.49	<.0007	.018
83-POSF-0973	86.54	13.89	<.0006	.024

NOTE: 1. The carbon, hydrogen, and nitrogen analyses were carried out by Schwarzkopf Laboratory using the Perkin-Elmer 240 Elemental Analyzer.

2. The oxygen analyses were carried out by Cintichem, Inc. using a neutron activation procedure.

TABLE 11. HEAT OF COMBUSTION DATA

Sample Number	(1)	Gross, BTU/lb (2)	Avg.	Net, BTU/lb[a]
82-POSF-0167	20,013	20,026	20,020	18,729
82-POSF-0168	19,860	19,814	19,837	18,470
82-POSF-0414	20,080	20,059	20,070	18,802
82-POSF-0417	20,084	20,044	20,064	18,727
82-POSF-0418	19,923	19,890	19,908	18,634
82-POSF-0477	20,045	20,058	20,052	18,794
83-POSF-0968	˙),299	19,328	19,314	18,075
83-POSF-0969	19,562	19,576	19,569	18,399
83-POSF-0970	20,002	20,043	20,002	18,689
83-POSF-0971	19,934	19,919	19,926	18,644
83-POSF-0972	19,349	19,364	19,356	18,217
83-POSF-0973	20,018	20,027	20,022	18,755

3. CHARACTERIZATION OF FUEL SAMPLES
AS PART OF A TYNDALL AFB PROGRAM

Six jet fuel samples (83-POSF-1557 through 83-POSF-1562), that were a part of the Tyndall AFB program, were characterized in detail. Analyses that were carried out were simulated distillation (ASTM D 2887-73), hydrocarbon type by mass spectrometry, gross heat of combustion (ASTM D 240-76), kinematic viscosity (ASTM D 445-79), density, true vapor pressure, and surface tension. The last four properties were determined as a function of temperature. Data are presented in Tables 12-15.

TABLE 12. SIMULATED DISTILLATION

% Recovered	83-POSF-1557 °C	83-POSF-1557 °F	83-POSF-1558 °C	83-POSF-1558 °F	83-POSF-1559 °C	83-POSF-1559 °F	83-POSF-1560 °C	83-POSF-1560 °F	83-POSF-1561 °C	83-POSF-1561 °F	83-POSF-1562 °C	83-POSF-1562 °F
0.5 (IBP)	32	89	29	85	117	242	109	228	150	302	149	300
1.0	39	102	34	94	129	264	126	258	159	317	158	317
5.0	62	144	57	134	168	335	170	338	172	341	171	341
10	71	160	67	153	186	367	190	374	178	353	178	352
20	92	197	89	192	203	396	206	403	186	367	187	368
30	104	219	98	208	215	420	218	424	197	386	197	387
40	123	253	117	242	224	435	226	439	206	403	206	402
50	144	291	139	281	232	449	233	452	213	415	214	418
60	169	336	168	335	240	464	242	467	224	435	225	437
70	195	383	198	388	246	476	248	479	230	446	232	449
80	217	423	217	423	258	496	260	500	241	466	244	471
90	238	461	238	460	270	518	273	523	255	492	260	500
95	253	461	254	489	279	535	283	542	268	514	274	525
99	276	529	286	547	306	583	310	590	300	573	303	577
99.5 (FBP)	285	545	295	562	315	599	316	600	312	593	310	590

TABLE 13. HYDROCARBON TYPE ANALYSES

	Weight percent									
	83-POSF-1557		83-POSF-1558		83-POSF-1559		83-POSF-1560		83-POSF-1561	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	60.3	54.8	64.6	59.8	45.4	45.8	46.4	46.5	44.2	44.5
Monocycloparaffins	20.8	-	17.7	-	35.8	-	35.5	-	33.1	-
Dicycloparaffins	3.2[d]	-	3.3[d]	-	1.4[d]	-	1.1[d]	-	0	-
Total cycloparaffins	24.0	24.2	21.0	20.6	37.2[d]	36.3	36.6	35.8	33.1[d]	31.3
Alkylbenzenes	12.3	18.3	11.5	17.2	8.5	10.3	8.5	10.5	11.5	15.1
Indans and tetralins	2.6	2.7	2.3	2.4	7.6	6.1	7.5	6.1	10.1	8.1
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	0.8	0	0.6	0	1.3	1.5	1.0	1.1	1.1	1.0
Average carbon no.	8.4		8.5		11.0		11.1		10.4	

	Weight percent	
	83-POSF-1562	
	ASTM[a]	Monsanto[b]
Paraffins	44.0	44.3
Monocycloparaffins	33.1	-[c]
Dicycloparaffins	0	-[c]
Total cycloparaffins	33.1[d]	31.2
Alkylbenzenes	11.5	15.2
Indans and tetralins	10.3	8.2
Indenes and dihydro-naphthalenes	-	0
Naphthalenes	1.1	1.1
Average carbon no.	10.4	

[a] Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b] Monsanto Method 21-PQ-38-63.

[c] Dash indicates method does not provide information on these specific compound categories.

[d] Sum of two preceding values.

TABLE 14. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Vapor pressure, (mm Hg)	Kinematic viscosity (centistokes)	Density, (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1557				
-30°F	6.3[a]	2.2738	0.7970	28.3[b]
32°F	35	1.2354	0.7706	25.3
59°F	-	-	0.7593	-
70°F	80	0.9298	0.7544	23.4
100°F	142	0.7718	0.7411	22.0
83-POSF-1558				
-30°F	5[a]	2.2839	0.7938	28.7[b]
32°F	30	1.2128	0.7673	25.7
59°F	-	-	0.7559	-
70°F	72	0.9505	0.7510	23.8
100°F	132	0.7940	0.7378	22.4
83-POSF-1559				
-30°F	4[a]	10.2026	0.8445	32.9[b]
32°F	11	3.5333	0.8204	29.6
59°F	-	-	0.8103	-
70°F	18	2.2231	0.8058	27.5
100°F	25	1.6667	0.7938	25.8
83-POSF-1560				
-30°F	1.8[a]	9.5565	0.8419	32.3[b]
32°F	6	3.3478	0.8178	29.3
59°F	-	-	0.8077	-
70°F	11	2.1392	0.8032	27.4
100°F	17	1.6300	0.7913	25.9
83-POSF-1561				
-30°F	-[c]	6.1559	0.8385	32.0
32°F	3.1	2.5446	0.8140	28.7
59°F	-	-	0.8036	-
70°F	6.6	1.6999	0.7990	26.7
100°F	11.2	1.3216	0.7868	25.2

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TABLE 14 (continued)

	Vapor pressure, (mm Hg)	Kinematic viscosity (centistokes)	Density, (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1562				
-30°F	-^{c}	6.1839	0.8384	31.2^b
32°F	3.1	2.5465	0.8149	28.3
59°F	-	-	0.8036	-
70°F	6.5	1.7082	0.7990	26.6
100°F	11.1	1.3340	0.7868	25.3

[a]Value determined by extrapolation of Log P versus 1/T vapor pressure relationship.

[b]Obtained by linear regression extrapolation of data.

[c]Values too low to be read from plot.

TABLE 15. HEAT OF COMBUSTION DATA

Sample number	Gross BTU/lb			Net BTU/lb[a]
	1	2	Avg.	
83-POSF-1557	20,074	20,088	20,081	20,081 - (91.23x%H)
83-POSF-1558	20,091	20,092	20,092	20,092 - (91.23x%H)
83-POSF-1559	19,885	19,872	19,879	19,879 - (91.23x%H)
83-POSF-1560	19,823	19,846	19,835	19,835 - (91.23x%H)
83-POSF-1561	19,811	19,782	19,797	19,797 - (91.23x%H)
83-POSF-1562	19,843	19,791	19,817	19,817 - (91.23x%H)

[a]The value for hydrogen content of the sample (% H) is not available at this time. The net heat of combustion can be calculated using this equation when % H is obtained.

4. CHARACTERIZATION OF JP-4 FUELS FROM CARIBOU SHALE OIL REFINING PROGRAM

Six fuels (83-POSF-1261, 83-POSF-1271, 83-POSF-1301, 83-POSF-1306, 83-POSF-1610 and 83-POSF-1612) were characterized in detail by means of simulated distillation (ASTM D 2887-73), gross heat of combustion (ASTM D 240-76), hydrocarbon type analyses, kinematic viscosity (ASTM D 445-79), density, true vapor pressure, and surface tension. The last four properties were determined as a function of temperature. Data are presented in Tables 16-19.

Specific heat and dielectric constant data were obtained on two of the fuels, and are contained in Tables 20 and 21 respectively. The specific heat analyses reported in Table 20 were carried out at Monsanto, St. Louis by differential scanning calorimetry (DSC). The instrumentation used was a Perkin-Elmer DSC-II C with an Intracooler II subambient accessory and a Thermal Analysis Data Station computer system for data acquisition and manipulation. A plot of the data output for fuel 83-POSF-1610 is shown in Figure 3.

The dielectric constant results given in Table 21 are the first shale-derived fuels analyzed here. The measurements, made at a frequency of 400 Hz, were carried out in a three terminal guarded cell relative to air at the same temperature. A General Radio 1615 A capacitance bridge and guard circuit were used in the determinations. Linear regression analyses were performed on the data to determine the dielectric constant as a linear function of temperature. A plot of dielectric constant results with fuel 83-POSF-1610 is shown in Figure 4.

TABLE 16. SIMULATED DISTILLATION

% Recovered	83-POSF-1261		83-POSF-1271		83-POSF-1301		83-POSF-1306		83-POSF-1610		83-POSF-1612	
	°C	°F	°C	°F	°C	°F	°C	°F	°C	°F	°C	°F
0.5 (IBP)	29	85	28	82	28	82	27	81	28	83	28	83
1.0	31	87	28	82	28	82	28	82	30	86	29	85
5.0	67	152	55	131	55	132	54	129	37	99	58	137
10	84	183	72	161	74	164	72	161	58	137	73	164
20	99	211	101	214	103	217	101	215	91	195	99	210
30	108	226	116	241	118	244	120	248	113	235	118	245
40	118	245	133	272	137	278	139	283	131	268	137	278
50	126	259	150	302	156	312	156	313	146	296	155	310
60	139	282	169	336	173	344	172	341	168	335	172	342
70	151	305	190	374	193	380	189	372	189	372	193	379
80	180	355	212	414	214	417	206	403	210	410	213	415
90	220	427	234	454	235	456	226	438	231	448	233	452
95	248	478	252	486	253	488	241	466	244	471	247	476
99	293	559	304	579	304	575	301	574	263	506	266	510
99.5 (FBP)	311	591	325	617	326	619	323	614	270	518	272	521

TABLE 17. HYDROCARBON TYPE ANALYSES

Weight percent

	83-POSF-1261		83-POSF-1271		83-POSF-1301		83-POSF-1306		83-POSF-1610	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	54.5	48.7	54.5	48.9	54.1	48.6	51.2	57.7	54.3	49.6
Monocycloparaffins	32.7	-	31.5	-	32.9	-	34.9	-	31.6	-
Dicycloparaffins	4.1[d]	-	3.6[d]	-	4.5[d]	-	5.0[d]	-	3.6[d]	-
Total cycloparaffins	36.8	38.6	35.1	37.0	37.4	38.4	39.9[d]	20.6	35.2[d]	37.4
Alkylbenzenes	8.0	12.4	8.9	14.1	8.0	12.6	7.6	19.6	8.0	11.9
Indans and tetralins	0.3	0.3	0.3	0	0.5	0.4	1.1	1.6	2.0	2.1
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0
Naphthalenes	0.2	0	1.3	0	0	0	0.2	0.5	0.5	0
Average carbon no.									8.6	

Weight percent

	83-POSF-1612	
	ASTM[a]	Monsanto[b]
Paraffins	54.0	48.6
Monocycloparaffins	32.0	-
Dicycloparaffins	3.9[d]	-
Total cycloparaffins	35.9[d]	37.4
Alkylbenzenes	7.9	11.9
Indans and tetralins	1.9	2.1
Indenes and dihydro-naphthalenes	-	0
Naphthalenes	0.3	0
Average carbon no.	8.6	

[a] Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b] Monsanto Method 21-PQ-28-63.

[c] Dash indicates method does not provide information on these specific compound categories.

[d] Sum of two preceding values.

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TABLE 18. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Vapor pressure, (mm Hg)	Kinematic viscosity (centistokes)	Density, (g/cm^3)	Surface tension (dynes/c)
83-POSF-1261				
-30.1°F	-	2.434	-	-[b]
-20°F	9[a]	2.141	0.7928	27.9[b]
-4°F	-	1.784	-	-
32°F	37	1.318	0.7721	25.4
59°F	-	-	0.7604	-
70°F	86	0.9798	0.7555	23.8
100°F	152	0.8082	0.7426	22.6
140°F	304	0.6478	0.7254	20.4
83-POSF-1271				
-30.1°F	-	2.285	-	-[b]
-20°F	8[a]	2.018	0.7943	28.1[b]
-4°F	-	1.684	-	-
32°F	35	1.251	0.7702	25.4
59°F	-	-	0.7583	-
70°F	84	0.9390	0.7534	23.5
100°F	155	0.7769	0.7405	22.0
140°F	320	0.6356	0.7232	19.9
83-POSF-1301				
-30.1°F	-	2.371	-	-[b]
-20°F	8[a]	2.095	0.7927	28.4[b]
-4°F	-	1.770	-	-
32°F	36	1.297	0.7716	25.7
59°F	-	-	0.7597	-
70°F	85	0.9753	0.7548	23.8
100°F	155	0.8042	0.7419	22.1
140°F	320	0.6447	0.7248	20.0
83-POSF-1306				
-30.1°F	-	2.387	-	-[b]
-20°F	10[a]	2.090	0.7941	28.6[b]
-4°F	-	1.754	-	-
32°F	41	1.294	0.7731	25.8
59°F	-	-	0.7613	-
70°F	92	0.9668	0.7565	23.9
100°F	167	0.7985	0.7435	22.3
140°F	330	0.6519	0.7259	20.1

TABLE 18 (continued)

	Vapor pressure, (mm Hg)	Kinematic viscosity (centistokes)	Density, (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1610				
-30.1°F	-	2.4189	-	-
-20°F	9[a]	2.1326	0.7944	28.3[b]
-4°F	-	1.7692	-	-
32°F	37	1.3171	0.7722	25.6
59°F	-	-	0.7606	-
70°F	83	0.9833	0.7559	23.6
100°F	147	0.8067	0.7431	22.0
140°F	295	0.6498	0.7260	19.9
83-POSF-1612				
-30.1°F	-	2.4263	-	-
-20°F	9[a]	2.1432	0.7955	28.7[b]
-4°F	-	1.7803	-	-
32°F	37	1.3294	0.7728	25.9
59°F	-	-	0.7610	-
70°F	84	0.9880	0.7562	23.9
100°F	150	0.8052	0.7431	22.3
140°F	300	0.6475	0.7256	20.2

[a]Value determined by extrapolation of Log P versus 1/T vapor pressure relationship.

[b]Obtained by linear regression extrapolaton of data.

TABLE 19. HEAT OF COMBUSTION DATA

Sample number	Gross BTU/lb 1	2	Avg.	Net BTU/lb[a]
83-POSF-1261	20,010	20,018	20,014	20,014 - (91.23 x %H)
83-POSF-1271	19,984	20,011	19,998	19,998 - (91.23 x %H)
83-POSF-1301	20,038	20,044	20,041	20,041 - (91.23 x %H)
83-POSF-1306	20,022	20,005	20,014	20,014 - (91.23 x %H)
83-POSF-1610	20,100	20,129	20,115	20,115 - (91.23 x %H)
83-POSF-1612	20,121	20,149	20,135	20,135 - (91.23 x %H)

[a] The value for hydrogen content of the sample (%H) is not available at this time. The net heat of combustion can be calculated using this equation when %H is obtained.

TABLE 20. SPECIFIC HEAT ANALYSES

Sample No.	Cal/g°C or BTU/lb°F 4°F (-15.6°C)	60°F (15.6°C)	100°F (37.8°C)
83-POSF-1610	0.440 ± .005	0.463 ± .007	0.485 ± .007
83-POSF-1612	0.434 ± .001	0.456 ± .001	0.483 ± .003
Heptane (>99% Purity)	0.508	0.533	0.563
Heptane (literature value)[a]	0.504	0.528	0.550

[a] n - Heptane literature value interpolated from API Project 44, Table 23-2 - (1.202) - VC.

TABLE 21. DIELECTRIC CONSTANTS

Sample	-20[a]	32	70	100	140
83-POSF-1610	2.092	2.060	2.037	2.018	1.993
83-POSF-1612	2.091	2.060	2.038	2.021	1.998

[a] Obtained by linear regression extrapolation of data.(°F)
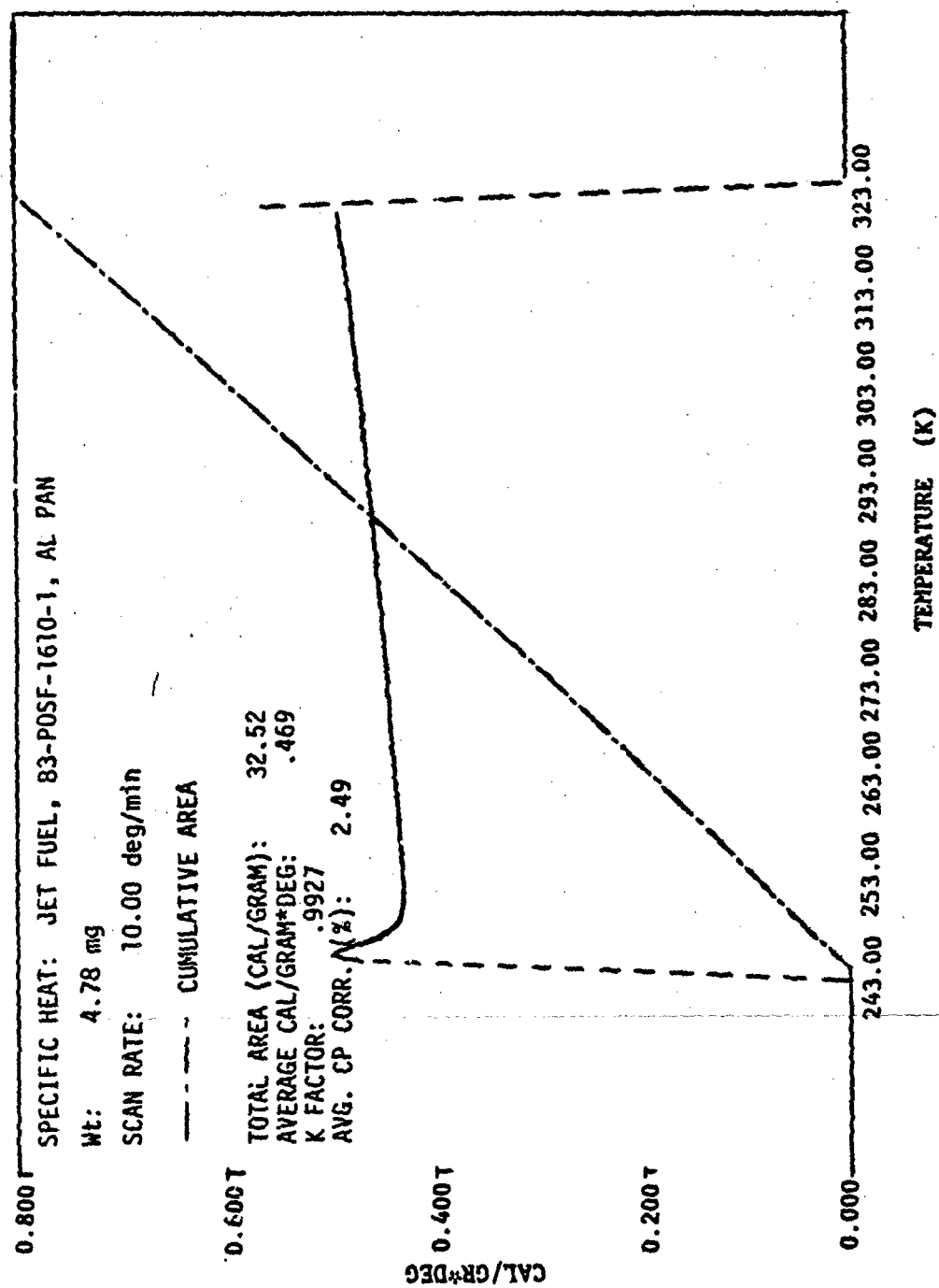
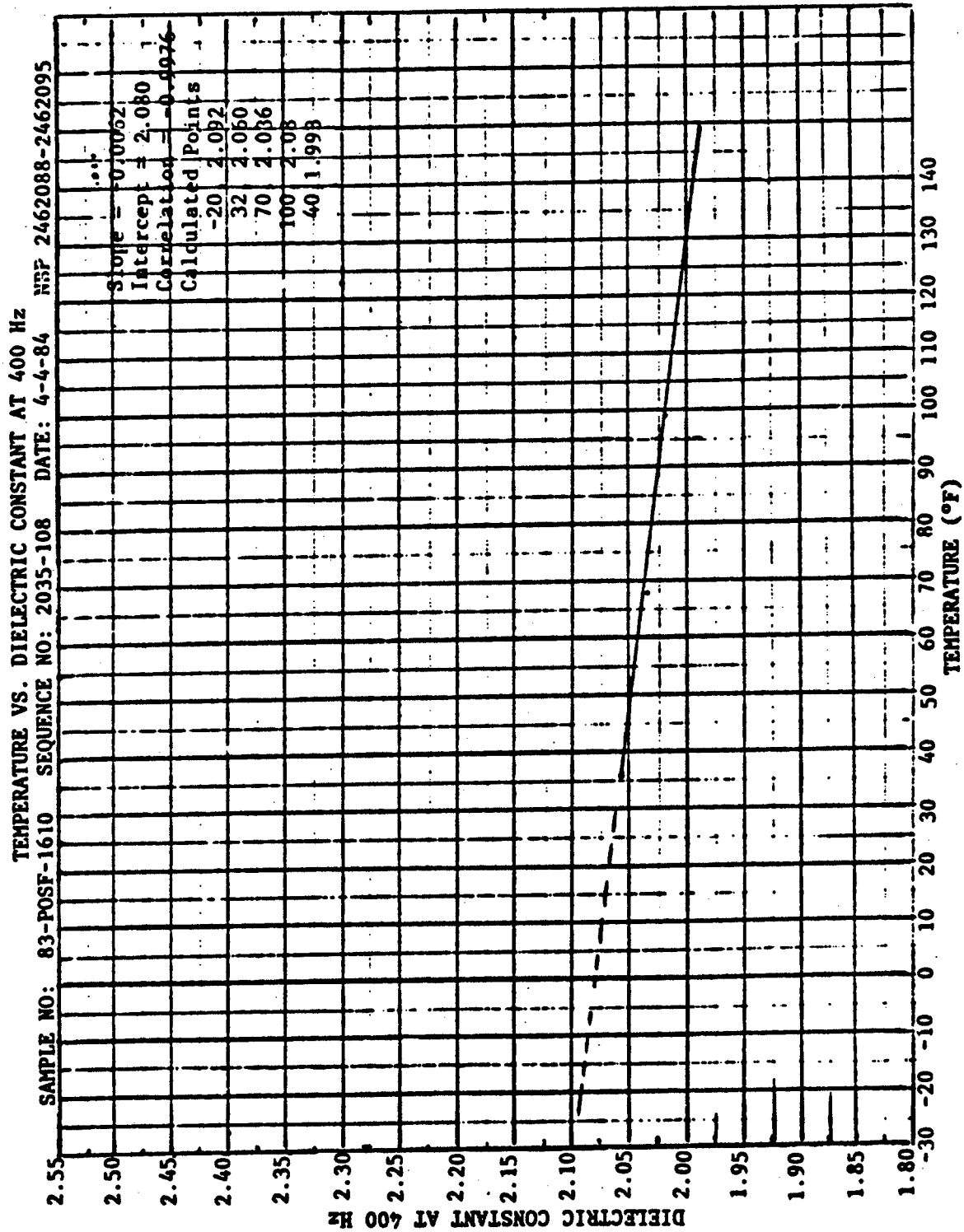Figure 3. Plot of Specific Heat vs. Temperature for Fuel 83-POSF-1610

SPECIFIC HEAT: JET FUEL, 83-POSF-1610-1, AL PAN

Wt: 4.78 mg

SCAN RATE: 10.00 deg/min

— – — CUMULATIVE AREA

TOTAL AREA (CAL/GRAM):   32.52
AVERAGE CAL/GRAM*DEG: .469
K FACTOR: .9927
AVG. CP CORR.(%):   2.49

CAL/GR*DEG

TEMPERATURE (K)

243.00 253.00 263.00 273.00 283.00 293.00 303.00 313.00 323.00

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Figure 4. Dielectric Constant vs. Temperature at 400 Hz

5. CHARACTERIZATION OF SHALE OIL PROCESS STREAMS FROM CARIBOU REFINERY

Two samples of hydrotreated shale oil from the Caribou refinery at Woods Cross, Utah have been characterized by the determination of composition and boiling range. The samples were coded as follows:

| 83-POSF-0979 | Second Pass Through Hydrotreater |
| 83-POSF-0980 | First Pass Through Hydrotreater |

Simulated distillation by gas chromatography (ASTM D 2889) was performed and results are presented in Table 22.

Carbon and hydrogen aromaticity, i.e., the atom ratio of aromatic carbon or hydrogen to total carbon or hydrogen, as appropriate, was determined by proton and carbon-13 Fourier transform NMR. These analyses were conducted on a Varian CFT-20 Fourier transform spectrometer containing a Varian 602L computer for data acquisition, data reduction, and system control. The spectrometer was operated at 20 megahertz for the ^{13}C analyses and 79.54 megahertz for the proton NMR analyses. The following instrument conditions were utilized for the two analyses:

Type NMR Analysis		Carbon-13 (^{13}C)	Proton (^1H)
Sample probe		8 mm	5 mm
Sweep Width		4000 Hz	1000 Hz
Number of Transients		1000	50
Acquisition Time		1.023 sec.	4.095 sec.
Pulse Width 90°		18 μsec.	30 μsec.
Pulse Delay		5 sec.	8 sec.
Homo-Spoil Time[a]		Not on	8 msec.
Data Points		8192	8192
Decoupler Mode		3[b]	--
Chemical Shift Regions	Aromatics	~150-110 ppm	8.3-6.5 ppm
for Integral Data	Aliphatics	~70-4 ppm	4.0-0.2 ppm

---

[a] Homospoil was on during pulse delay.

[b] The gated proton decoupler was on during acquisition and off during delay.

Integral data and aromaticities are presented in Table 23, and NMR spectra of the samples are shown in Figures 5-8.

Hydrocarbon type analyses were conducted by mass spectrometry. Because of the wide boiling range of the samples, each was separated into two fractions by simple distillation. The cut points of the lower boiling fractions were selected so that the boiling range was suitable for analysis by ASTM Method D 2789. The higher boiling materials were further separated into aromatic and nonaromatic fractions using the ASTM D 2549 procedure. These fractions were then analyzed by the D 2425 mass spectrometric hydrocarbon type analysis method. The analytical results presented in Table 24 consist of composited values which were computed from the compositions and quantities of each of three fractions.

Since preliminary data were required on these samples on a more immediate basis, thus initial hydrocarbon type analyses were conducted by the Monsanto 21-PQ-38-63 method. Results of these analyses, which essentially can be only approximations on this kind of sample, are included in Table 24.

TABLE 22. SIMULATED DISTILLATION

% Recovered	83-POSF-0979 °C	°F	83-POSF-0980 °C	°F
0.5 (IBP)	116	241	80	176
1	126	258	93	199
5	161	322	138	280
10	180	356	166	330
20	209	408	193	399
30	226	439	222	431
40	240	464	240	464
50	257	494	259	498
60	274	524	280	537
70	295	563	300	572
80	313	595	325	618
90	346	656	362	684
95	373	704	393	740
99	428	802	440	824
99.5 (FBP)	439	822	450	842

TABLE 23. INTEGRATED AREAS[a] AND CALCULATED AROMATICITIES FROM CARBON-13 AND PROTON NMR

	83-POSF-0979	83-POSF-0980
Carbon-13		
Aromatic Region, 110-150 ppm (CAR)	10	18
Aliphatic Region, 4-70 ppm	84.25	82
Total (C_T)	94.25	100
Carbon Aromaticity (CAR/C_T)	0.11	0.18
Proton		
Aromatic Region, 6.6-8.3 ppm (HAR)	6	7
Aliphatic Region, 0.5-4.0 ppm	197	175
Total (H_T)	203	182
Hydrogen Aromaticity (HAR/H_T)	0.03	0.04

[a]Average of four integrations.
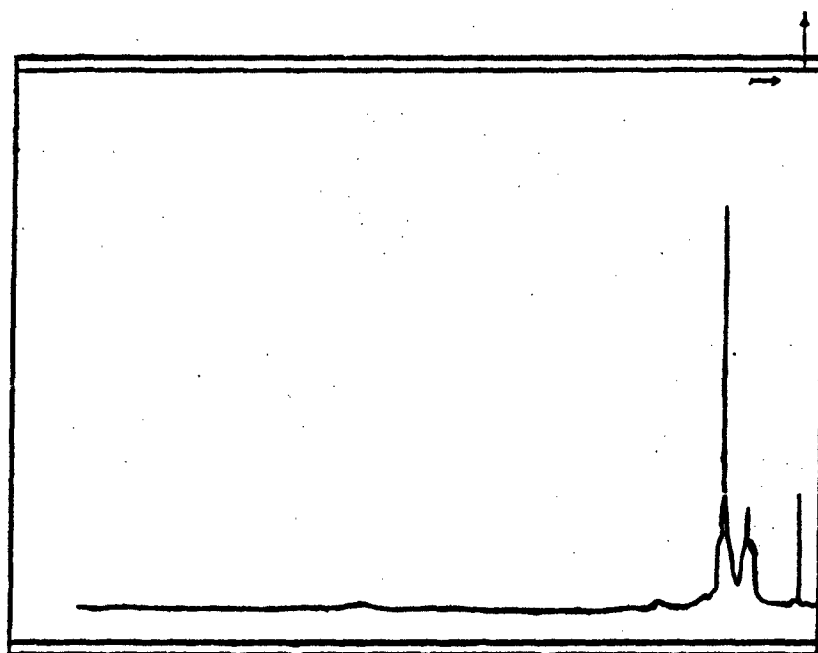
Sample 0990.
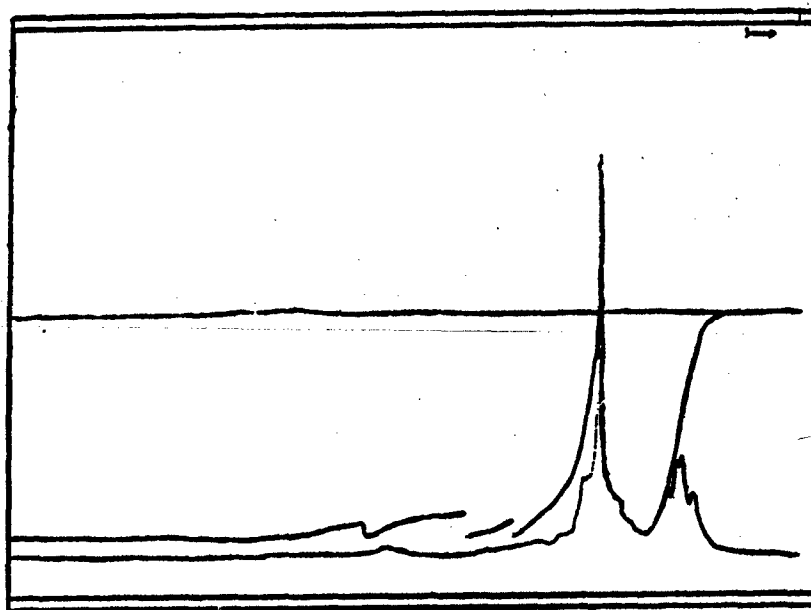
Sample 0980

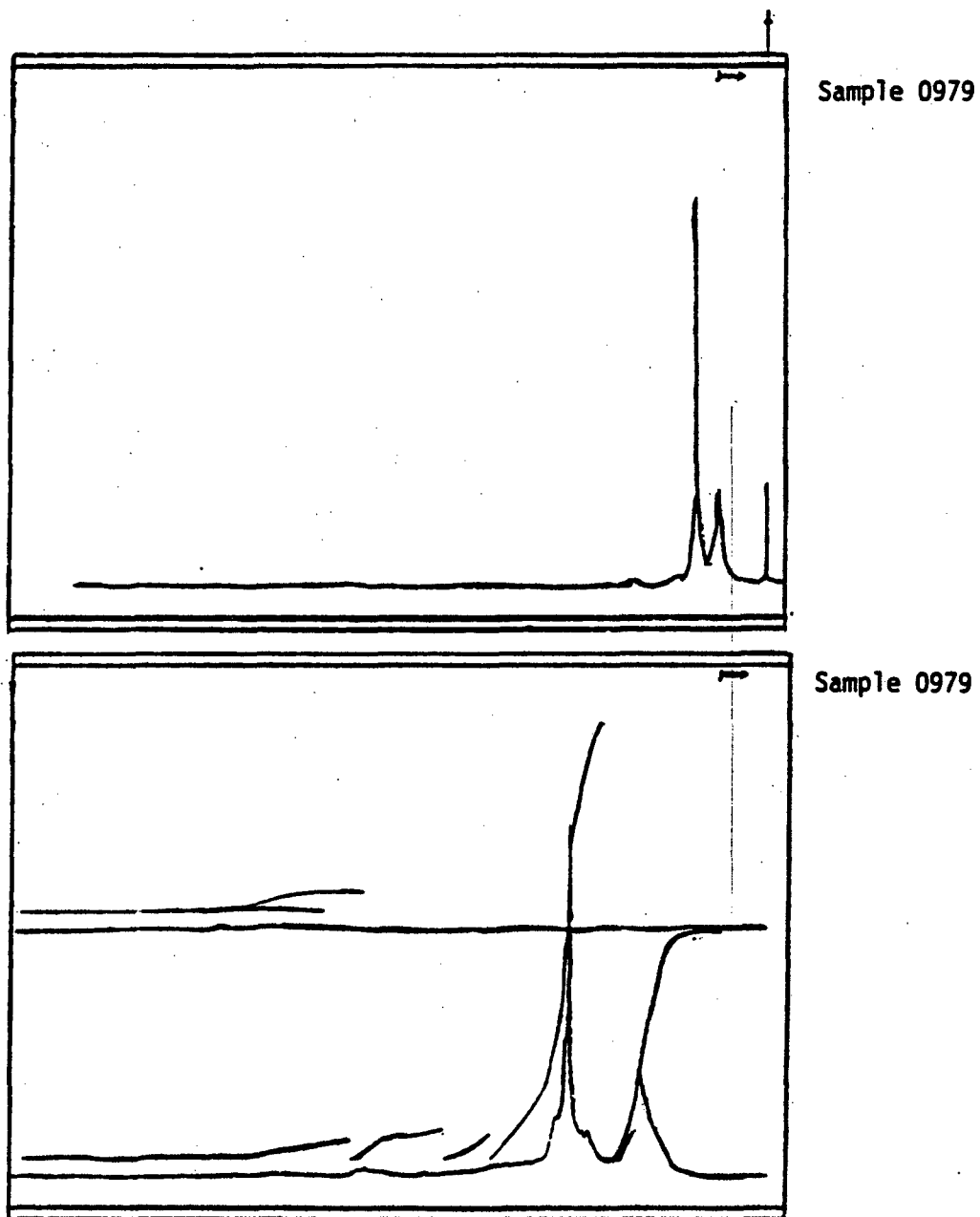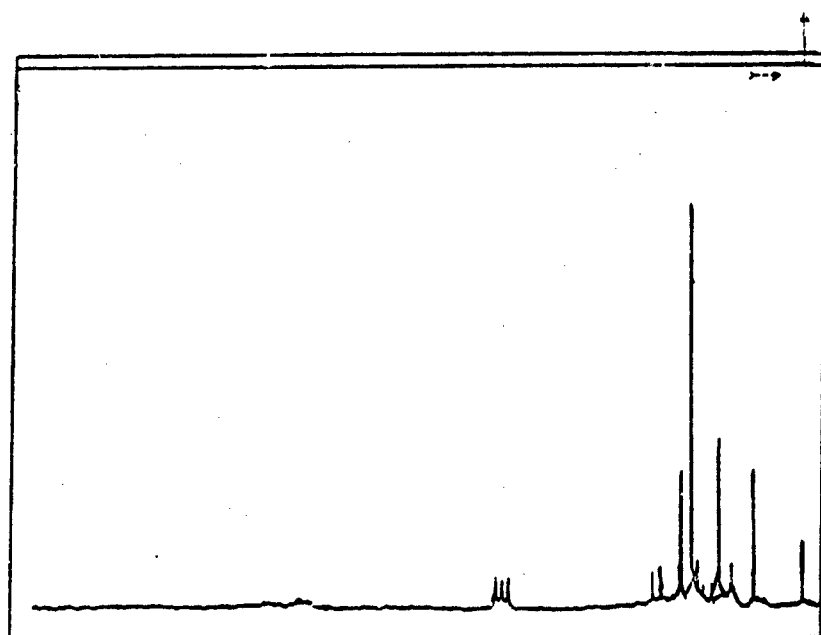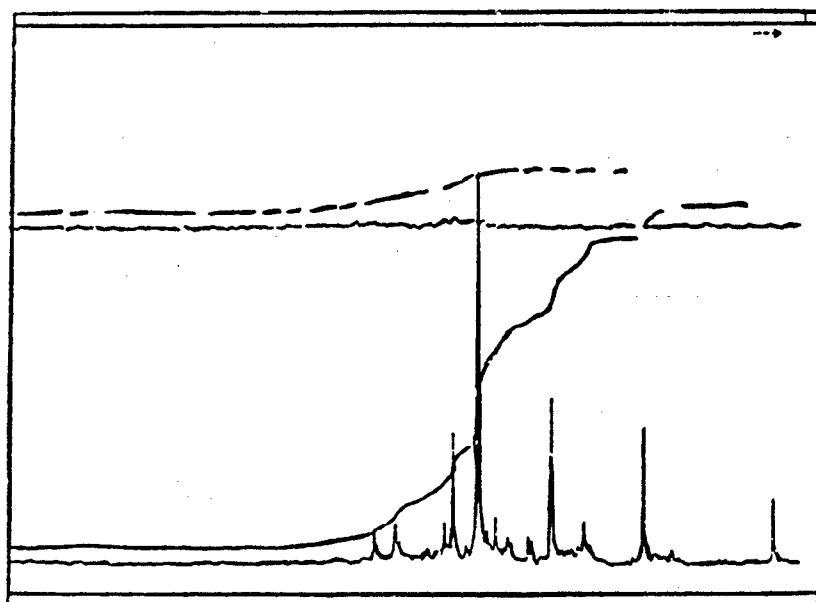Figure 5.   Fuel 83-POSF-0980 ^{1}H spectra.

Sample 0979

Sample 0979

Figure 6.   Fuel 83-POSF-0979 ^{1}H spectra.

Sample 0979

Sample 0979

Figure 7.   Fuel 83-POSF-0979 ^{13}C spectra.

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Sample 0980

Sample 0980
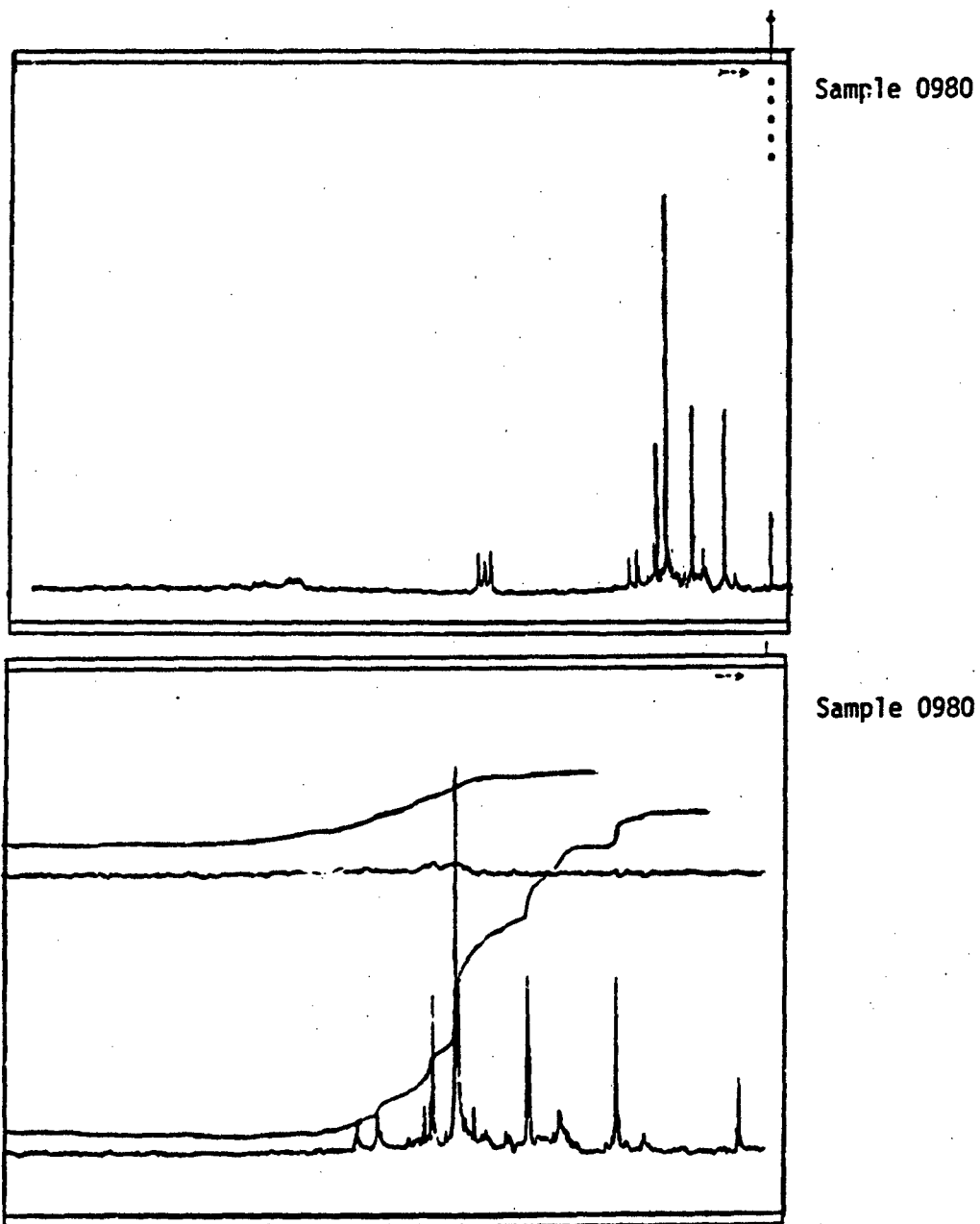
Figure 4.   Fuel 83-POSF-0980 ^{13}C spectra.

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TABLE 24.   HYDROCARBON TYPE ANALYSES

	83-POSF-0979		83-POSF-0980	
	ASTM D 2425[a] and D 2789	Monsanto[b] 21-PQ-36-63	ASTM D 2425[a] and D 2789	Monsanto[b] 21-PQ-36-63
Paraffins	37.8	48.2[c]	37.5	48.7[c]
Monacycloparaffins	32.8	33.1[c]	30.2	28.5[c]
Dicycloparaffins	6.1	–	2.4	–
Tricycloparaffins	1.5	–	0.8	–
Alkylbenzenes	12.0	9.7	13.8	11.3
Indans & Tetralins	6.6	6.6	8.5	7.8
Indenes	0.7	0.4	0.6	0.1
Naphthalenes	1.6	2.0	4.3	3.6
Acenaphthenes	0.4	–	0.8	–
Acenaphthylenes	0.3	–	0.7	–
Tricyclic Aromatics	0.2	–	0.4	–

[a] Samples separated into higher and lower boiling fractions to which D 2549/2425 methods and D 2789 method were applied; respectively. Results were combined as shown.

[b] Monsanto method used for preliminary analysis though the broad boiling range makes the analysis an approximate one.

[c] Total measured cycloparaffins.

[d] Dashes indicate this compound class cannot be determined by method.

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Four samples of shale oil feedstock from the Caribou Refinery at
Woods Cross, Utah (83-POSF-1285, 83-POSF-1286, 83-POSF-1300, and
83-POSF-1308) and one bottoms sample (83-POSF-1310) were analyzed
for carbon, hydrogen, and oxygen content; one sample, 83-POSF-1308
had a nitrogen determination and another sample 83-POSF-1310, had
a nitrogen and sulfur analysis made on it. In addition, samples
83-POSF-1285 and -1286 were characterized as to carbon and hydroge
aromaticity by me ns of proton and carbon-13 Fourier transform
NMR.

The samples being discussed here were identified as follows:

83-POSF-1285	Caribou distilled shale oil, Batch 1
83-POSF-1286	Caribou raw shale oil, Batch 2
83-POSF-1300	Caribou distilled shale oil, Batch 2
83-POSF-1303	Caribou distilled shale oil, Batch 2
83-POSF-1310	Caribou shale oil bottoms

The carbon, hydrogen, nitrogen, and oxygen analyses are compiled
in Table 25, and the carbon and hydrogen aromaticities in Table 2

In examining the oxygen data, it can be seen that the oxygen level
in the distilled shale oil from Batch 1 is substantially higher
than the distilled shale oil samples from Batch 2. Whether this
was reflected in the difficulty of hydrotreating the two batches
is unknown to us, as both batches were reported to have required
excessive hydrogen for this processing step.

The NMR analyses were conducted in a manner similar to that dis-
cussed previously in this section. The equipment utilized was a
Varian CFT-20 Fourier transform spectrometer containing a Varian
602L computer for data acquisition, data reduction, and system
control. The NMR results reflect a slight batch-to-batch variatio
with the shale feedstocks.

TABLE 25. ELEMENTAL ANALYSIS

Sample No.	Carbon, wt. %	Hydrogen, wt. %	Oxygen, wt. %	Nitrogen, wt. %	Sulfur, wt. %
83-POSF-1285	85.22	12.45	1.31	-	-
83-POSF-1286	78.69	11.72	6.79	-	-
83-POSF-1300	85.66	11.96	0.75	-	-
83-POSF-1308	86.16	13.29	0.76	0.44	-
83-POSF-1310	86.19	12.53	0.89	1.31	0.55

NOTE: (1) All oxygen determinations made by Cintichem, Inc., (2) C, H, N, and S on Nos. 1308 and 1310 by Schwarzkopf Laboratory, and (3) C and H on Nos. 1285, 1286, and 1300 by Galbraith Laboratories, Inc.

TABLE 26. AROMATICITIES CALCULATED FROM INTEGRATED AREAS DETERMINED BY CARBON-13 AND PROTON NMR

	83-POSF-1285	83-POSF-1286
Carbon-13		
Aromatic Region, 110-150 ppm (C_{ar})	18	18
Aliphatic Region, 4-70 ppm	65	61
Total (C_T)	83	79
Carbon Aromaticity (C_{ar}/C_T)	0.217	0.228
Proton		
Aromatic Region, 6.6-8.3 ppm (H_{ar})	11	5
Aliphatic Region, 0.5-4.0 ppm	272	116
Total (H_T)	283	121
Hydrogen Aromaticity (H_{ar}/H_T)	0.039	0.41

6. CHARACTERIZATION OF SAMPLES FROM THE HIGH DENSITY FUELS PROGRAM

Three experimental high density jet fuel samples were character-
ized. These fuel samples which were described as "light pyrolysis
fuel oil" had the following sample numbers: 82-POSF-0161, 83-POSF-
0800, and 84-POSF-1758. Analyses carried out on these fuels were
kinematic viscosity, density, surface tension, and dielectric con-
stant. Results are compiled in Tables 27 and 28.

The dielectric constant measurements were made at a frequency of
400 Hz and were carried out in a three terminal guarded cell rela-
tive to air at the same temperature. A general Radio 1615A capa-
citance bridge and guard circuit were used in the determinations.
Linear regression analyses were performed on the data to determine
the dielectric constant as a linear function of temperature.

Another sample of light pyrolysis fuel oil from the high density
fuels program (84-POSF-1949) was characterized. Analyses conducted
were hydrocarbon type by mass spectrometry, simulated distillation,
heat of combustion, surface tension, density, and kinematic vis-
cosity. The last three analyses were carried out as a function of
temperature. Results are given in Tables 29-32. A qualification
is made with regard to the hydrocarbon type analysis. It is not
known how precise the results are, since the method is not applic-
able for a sample that is predominantly one compound type, such as
dicycloparaffin in this case.

Analysis of a carbon slurry fuel (83-POSF-1275) was made for den-
sity (D 1217-81) and gross heat of combustion (D 240-76). Results
are compiled in Table 33.

The carbon slurry fuel posed severe sampling problems because it
was quite viscous. Accordingly, the slurry was shaken vigorously
before sampling in order to get as homogeneous a sample as pos-
sible. Subsequent transfers of the slurry to the capsule of the

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combustion bomb and to the body of the pycnometer were achieved with some difficulty. Nonetheless, repeatability of the heat of combustion results was excellent.

TABLE 27. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
82-POSF-0161			
-30°F	15.451	-	35.8[a]
32°F	4.889	0.8844	32.8
59°F	-	0.8737	-
70°F	2.973	0.8694	30.9
100°F	2.178	0.8575	29.5
83-POSF-0800			
-30°F	16.843	-	37.1[a]
32°F	4.914	0.9067	33.8
59°F	-	0.8961	-
70°F	2.911	0.8921	31.8
100°F	2.123	0.8807	30.2
84-POSF-1758			
-30°F	16.012	-	36.0[a]
32°F	4.880	0.8957	33.0
59°F	-	0.8849	-
70°F	2.940	0.8807	31.2
100°F	2.129	0.8688	29.8

[a] Value determined by linear regression extrapolation of data.

TABLE 28. DIELECTRIC CONSTANTS @ 400 Hz

Sample No.	Temperature, °F			
	-32[b]	32	70	100
82-POSF-0161	2.216	2.181	2.160	2.143
83-POSF-0800	2.333	2.297	2.274	2.256
84-POSF-1758	2.278	2.241	2.218	2.200

[b] Value determined by linear regression extrapolation of data.

TABLE 29.   SIMULATED DISTILLATION

% Recovered	84-POSF-1949	
	°C	°F
0.5 (IBP)	116	241
1.0	132	270
5.0	182	360
10	186	367
20	196	385
30	202	396
40	208	406
50	214	417
60	221	430
70	229	444
80	238	460
90	251	484
95	259	498
99	272	522
99.5 (FBP)	276	529

TABLE 30.   HYDROCARBON TYPE ANALYSES

	Weight Percent 84-POSF-1949	
	ASTM[a]	Monsanto[b]
Paraffins	3.3	0
Monocycloparaffins	0	-
Dicycloparaffins	88.3	-
Total cycloparaffins	88.3[d]	93.6
Alkylbenzenes	5.1	3.2
Indans & Tetralins	3.0	2.4
Indenes & dihydro- naphthalenes	-	0
Naphthalenes	0.3	0.8
Average carbon number	~12	

[a]Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b]Monsanto Method 21-PQ-38-63.

[c]Dash indicates method does not provide information on these specific compound categories.

[d]Sum of two preceding values.

TABLE 31.   PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
84-POSF-1949			
-30.1°F	12.9078	0.9069	36.3[a]
32°F	4.4033	0.8830	33.0
70°F	2.7346	0.8683	31.0
100°F	2.0259	0.8567	29.4

[a]Value determined by linear regression extrapolation of data.

TABLE 32.   HEAT OF COMBUSTION

Sample Number	Gross, Btu/lb.			Net, Btu/lb.
	(1)	(2)	(Avg.)	
84-POSF-1949	19,497	19,462	19,480	19,480 - (91.23 x %H)

TABLE 33.   DENSITY AND HEAT OF COMBUSTION DATA

Sample No.	Density, g/mL at 15°C	Gross heat of combustion, Btu/lb			Net heat of combustion, Btu/lb[a]
		(1)	(2)	Average	
83-POSF-1275	1.2955	16,609	16,620	16,614	16,614 - (91.23 x % H)

[a]The value for the hydrogen content of the carbon slurry sample (% H) is not available to us at this time. The net heat of combustion can be calculated when the % H is obtained.

7. OTHER CHARACTERIZATIONS PROVIDED FOR AIR FORCE PROJECT SUPPORT

Samples examined by various methods of testing are listed as follows:

Sample Number	Type of Fuel
83-POSF-0841	JP-4
83-POSF-0842	JP-5
83-POSF-1064	JP-4
83-POSF-1083	JP-4
83-POSF-1183	Diesel Fuel
83-POSF-1274	JP-5/JP-8 Blend
83-POSF-1405	Experimental Jet Fuel
83-POSF-1406	Experimental Jet Fuel
84-POSF-2074	JP-4

The first five fuels were characterized in detail by means of simulated distillation (ASTM D 2887), gross heat of combustion (ASTM D 240-76), hydrocarbon-type analyses, kinematic viscosity (ASTM D 445-79), density, true vapor pressure and surface tension. The last four properties were determined as a function of temperature. Data are presented in Tables 34-37.

TABLE 34. SIMULATED DISTILLATION

% Recovered	83-POSF-0841 °C	°F	83-POSF-0842 °C	°F	83-POSF-1064 °C	°F	83-POSF-1083 °C	°F	83-POSF-1183 °C	°F
0.5 (IBP)	16	61	98	208	26	79	34	92	132	270
1.0	35	94	115	240	28	82	35	94	146	295
5.0	81	177	165	328	66	151	59	138	186	366
10	97	206	179	354	88	190	74	166	198	388
20	110	230	193	379	108	226	111	232	207	405
30	119	246	202	395	129	264	143	290	221	430
40	132	270	210	410	142	288	159	319	230	447
50	141	286	219	426	154	309	162	324	247	477
60	152	306	227	441	165	329	165	330	261	503
70	165	330	236	457	174	345	174	344	280	536
80	178	352	249	479	190	374	206	402	302	576
90	195	384	264	507	212	414	237	459	330	626
95	216	420	277	530	244	471	258	496	350	662
99	265	510	305	581	277	531	293	559	379	713
99.5 (FBP)	278	533	314	597	289	552	304	579	384	723

TABLE 35. HYDROCARBON TYPE ANALYSES

	Weight percent									
	83-POSF-0841		83-POSF-0842		83-POSF-1064		83-POSF-1083		83-POSF-1183	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	39.2	31.3	34.5	35.8	50.8	42.4	34.6	25.6	31.4	30.9
Monocycloparaffins	37.0	-	34.8	-	30.6	-	13.8	-	23.2	-
Dicycloparaffins	4.7[d]	-	4.8[d]	-	3.5[d]	-	1.8[d]	-	0.8[d]	-
Total cycloparaffins	41.7	43.7	39.6	39.3	34.1	36.5	15.6	13.4	24.0	22.8
Alkylbenzenes	18.1	25.0	9.7	12.0	14.5	21.1	45.7	59.2	12.3	15.5
Indans and tetralins	0.6	0	13.8	10.4	0.1	0	3.3	1.2	11.2	8.4
Indenes and dihydro-naphthalenes	-	0	-	0	-	0	-	0	-	0.7
Naphthalenes	0.4	0	2.4	2.5	0.5	0	0.8	0.6	21.1	21.7

[a] Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b] Monsanto Method 21-PQ-38-63.

[c] Dash indicates method does not provide information on these specific compound categories.

[d] Sum of two preceding values.

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TABLE 36. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
83-POSF-0841				
-30.1°F	-	2.046	-	-
-20°F	6[a]	1.819	0.8065	29.2[b]
-4°F	-	1.540	-	-
32°F	24	1.168	0.7856	26.6
59°F	-	-	0.7735	-
70°F	59	0.8810	0.7691	24.7
100°F	108	0.7308	0.7558	23.2
140°F	217	-	-	-
83-POSF-0842				
-30.1°F	-	9.000	-	-
-20°F	3[a]	7.238	0.8539	33.7[b]
4°F	-	5.306	-	-
32°F	7	3.215	0.8345	30.2
59°F	-	-	0.8234	-
70°F	14	2.063	0.8190	27.6
100°F	21	1.558	0.8071	25.5
140°F	36	-	-	-
83-POSF-1064				
-30.1°F	-	2.099	-	-
-20°F	8[a]	1.855	0.7963	29.6[b]
-4°F	-	1.588	-	-
32°F	33	1.189	0.7747	26.7
59°F	-	-	0.7627	-
70°F	78	0.8916	0.7577	24.6
100°F	141	0.7410	0.7442	22.9
140°F	278	-	-	-
83-POSF-1083				
-30.1°F	-	2.058	-	-
-20°F	5.5	1.835	0.8337	28.7
-4°F	-	1.548	-	-
32°F	25	1.142	0.8113	26.2
59°F	-	-	0.7996	-
70°F	60	0.8730	0.7948	24.5
100°F	114	0.7215	0.7819	23.0
140°F	234	-	-	-

TABLE 36 (continued)

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm^3)	Surface tension (dynes/cm)
83-POSF-1183				
-30.1°F	-	Frozen	-	-
-20°F	2^a	Frozen	0.9006	-
-4°F	-	10.277	-	-
32°F	5	5.252	0.8831	32.0
59°F	-	-	0.8721	-
70°F	9	2.998	0.8677	30.0
100°F	13.5	2.147	0.8560	28.4
140°F	23	-	-	-

^{a}Value determined by extrapolation of Log P versus 1/T vapor pressure relationship.

^{b}Obtained by linear regression extrapolation of data.

TABLE 37. HEAT OF COMBUSTION DATA

Sample number	Gross BTU/lb			Net BTU/lb^a
	1	2	Avg.	
83-POSF-0841	19,888	19,874	19,881	19,881 - (91.23 x %H)
83-POSF-0842	19,636	19,672	19,654	19,654 - (91.23 x %H)
83-POSF-1064	19,877	19,836	19,857	19,857 - (91.23 x %H)
83-POSF-1083	19,480	19,450	19,465	19,465 - (91.23 x %H)
83-POSF-1183	19,072	19,118	19,095	19,095 - (91.23 x %H)

^{a}The value for hydrogen content of the sample (%H) is not available at this time. Net heat of combustion can be calculated by this equation when %H is obtained.

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A blend of JP-5/JP-8 (83-POSF-1274) was characterized in detail by means of simulated distillation (ASTM D 2887), hydrocarbon type analysis, kinematic viscosity (ASTM D 445-79), and density. The last two properties were measured as a function of temperature. Data are presented in Tables 38-40.

TABLE 38. SIMULATED DISTILLATION

Percent recovered	83-POSF-1274 °C	83-POSF-1274 °F
0.5 (IBP)	38	101
1.0	113	236
5.0	153	307
10	166	331
20	184	364
30	197	386
40	207	405
50	217	422
60	227	440
70	236	457
80	249	481
90	265	509
95	281	538
99	336	637
99.5 (FBP)	348	658

TABLE 39. HYDROCARBON TYPE ANALYSES

	Weight percent 83-POSF-1274	
	ASTM[a]	Monsanto[b]
Paraffins	43.8	43.3
Monocycloparaffins	38.1	-
Dicycloparaffins	1.9	-
Total cycloparaffins	40.0[d]	38.4
Alkylbenzenes	9.9	11.8
Indans and tetralins	3.9	3.3
Indenes and dihydronaphthalenes	-	0
Naphthalenes	2.4	2.2
Average Carbon No.	10.5	

[a]Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b]Monsanto Method 21-PQ-38-63.

[c]Dash indicates that method does not provide information on this specific compound category.

[d]Sum of two preceding values.

TABLE 40. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Kinematic viscosity (centistokes)	Density (g/cm³)
83-POSF-1274		
-20°F	6.738	0.8403[a]
-4°F	4.870	-[a]
32°F	2.998[a]	0.8202
59°F	-[a]	0.8094
70°F	1.967	0.8049
100°F	1.492	0.7926
140°F	1.097	0.7769

[a]Data at these temperatures not obtained.

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Two experimental jet fuels (83-POSF-1405 and 83-POSF-1406) have been characterized in detail by means of simulated distillation (ASTM D 2887-73), hydrocarbon type analyses, density, and gross heat of combuston (ASTM D 240-76). Data from these analyses are presented in Tables 41-43.

TABLE 41.   SIMULATED DISTILLATION

Percent recovered	83-POSF-1405 °C	83-POSF-1405 °F	83-POSF-1406 °C	83-POSF-1406 °F
0.5 (IBP)	27	81	99	210
1.0	27	81	116	242
5.0	30	85	133	271
10	54	129	145	293
20	61	142	153	308
30	80	176	167	333
40	93	199	175	347
50	101	213	186	367
60	112	234	196	385
70	118	245	206	404
80	135	275	217	423
90	142	288	234	454
95	147	297	252	485
99	170	339	314	597
99.5 (FBP)	215	419	330	626

TABLE 42.   HYDROCARBON TYPE ANALYSES

	Weight percent			
	83-POSF-1405		83-POSF-1406	
	ASTM[a]	Monsanto[b]	ASTM[a]	Monsanto[b]
Paraffins	69.5	53.9	51.4	48.3
Monocycloparaffins	12.5	-	39.4	-
Dicycloparaffins	0.4	-	1.3	-
Total cycloparaffins	12.9[d]	13.0	40.7[d]	42.6
Alkylbenzenes	17.3	33.1	5.6	6.9
Indans and tetralins	0.3	0	1.2	1.1
Indenes and dihydronaphthalenes	-	0	-	0
Naphthalenes	0	0	1.1	1.1
Average Carbon No.	6.5		10.2	

[a]Modification of ASTM Method D 2789, values converted from volume percent using relative densities.

[b]Monsanto Method 21-PQ-38-63.

[c]Dash indicates that method does not provide informaton on these specific compound categories.

[d]Sum of two preceding values.

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TABLE 43. DENSITY AND HEAT OF COMBUSTION DATA

Sample no.	Density g/mL at 15°C	Btu/lb (1)	Btu/lb (2)	Average	Net heat of combustion Btu/lb[a]
83-POSF-1405	0.7255	20,164	20,147	20,156	20,156 - (91.23 x %H)
83-POSF-1406	0.7828	20,070	20,057	20.064	20,064 - (91.23 x %H)

[a]The value for hydrogen content of the sample (%H) is not available at this time. The net heat of combustion can be calculated using this equation when %H is obtained.

A sample of JP-4 fuel from the F109 Engine Test Program (84-POSF-2074) was characterized. Analyses conducted were heat of combustion, kinematic viscosity, density, true vapor pressure, and surface tension. The last four analyses were carried out as a function of temperature. Data are presented in Tables 44 and 45.

TABLE 44. PHYSICAL PROPERTIES AS A FUNCTION OF TEMPERATURE

	Vapor pressure (mm Hg)	Kinematic viscosity (centistokes)	Density (g/cm³)	Surface tension (dynes/cm)
84-POSF-2074				
-20°F	9[a]	1.7325	0.7928	27.9[b]
32°F	35	1.1116	0.7703	25.1
59°F	-	-	0.7587	-
70°F	79	0.8420	0.7539	23.3
100°F	139	0.7087	0.7409	21.4
140°F	280	0.5724	0.7236	19.5

[a]Value determined by extrapolation of Log P versus 1/T vapor pressure relationship.

[b]Obtained by linear regression extrapolation data.

TABLE 45. HEAT OF COMBUSTION DATA

Sample No.	Gross, Btu/lb (1)	Gross, Btu/lb (2)	Gross, Btu/lb (avg)	Net, Btu/lb[a]
84-POSF-2074	20,023	20,000	20,012	20,012-(91.23 x % H)

[a]The value for hydrogen content of the sample (% H) is not available at this time. The net heat of combustion can be calculated using this equation when % H is obtained.

SECTION III

ANALYSIS OF FUELS-RELATED DEPOSITS, CONTAMINANTS, AND IMPURITIES

Although stringent specifications have been established for all Air Force fuels, the quality of an "on-spec" fuel may be altered in a variety of ways during handling and transportation. Any time a fuel is transported, or even exposed to its surroundings, dust or particulate matter may be picked up, volatiles may be lost, trace metals dissolved from metal surfaces, or gums formed during storage.

Despite careful attention given to ensuring fuel integrity, situations may arise where a fuel has been contaminated with dissimilar fuels, or other organic materials. Aviation turbine fuels such as JP-4 may be shipped through the same pipeline as gasoline. Contamination of the jet fuel with even very low levels of gasoline containing tetraethyllead can have deleterious effects on the thermal stability of the jet fuel. Further, inadvertent mixing of two jet fuels, e.g., JP-7 with some amount of JP-4, could result in a fuel "off-spec" in volatility. Another form of contamination might be the introduction of elastomeric materials from seals or hoses, or material leached from poorly cured epoxy tank liners. When aircraft fuels come into contact with metal surfaces, they may acquire trace levels of metals in the form of oxides, inorganic salts, or organometallic compounds including naphthenates. Solubility of these metal compounds in fuel is a matter of concern because trace levels have been found to catalyze the high temperature degradation of hydrocarbon fuels.

During the course of this program, several investigations have been conducted to determine the nature and possible source of fuel particulate contaminants and fuel deposits.

1. IDENTIFICATION OF TURBINE BLADE CONTAMINANTS

The purpose of this task was to determine the composition of an orange deposit which had formed on the turbine blade of an Air Force TF 30 engine. Techniques used in making this determination were Electron Spectroscopy for Chemical Analysis (ESCA), and Energy Dispersive X-Ray Analysis (EDXA). These analyses were carried out at the University of Dayton Research Institute on the turbine blade identified as #33.

The EDXA spectra of the bulk alloy, and the orange deposit are shown in Figure 9. The chief constituents seen in the bulk alloy are cobalt, c romium, tungsten, and tantalum. When observing the orange deposit, the EDXA spectrum showed aluminum, iron, calcium, zinc, and ph sphorus being present along with cobalt and chromium, the major constituents of the alloy.

ESCA scans were recorded on the orange deposit before and after argon ion-sputtering the sample for one minute. (Actually, less than 100 $\overset{\circ}{A}$ of material are removed from the surface of the sample by this brief sputter.) Comparison of the two scans, Figures 10 and 11, shows that the carbon signal is reduced substantially after sputtering. This indicates that surface hydrocarbon contamination is being removed from the deposit. The ESCA scan taken after sputtering shows that iron, oxygen, calcium, zinc, phosphorus, and aluminum are detected in addition to carbon.

From an accurate measurement of the peak binding energies in the ESCA scan, one can infer the possible chemical states of the elements present by comparison with known reference values. From Figure 10, the following inferences can be drawn: (1) iron is most likely present as $Fe_2O_3 \cdot$ x H_2O, or as ferric phosphate; (2) calcium, as CaO, $CaCO_3$ or calcium phosphate; (3) phosphorus as $(PO_4)^{3}$; or $(PO_3)^{3}$; and (4) aluminum, as aluminum oxide.

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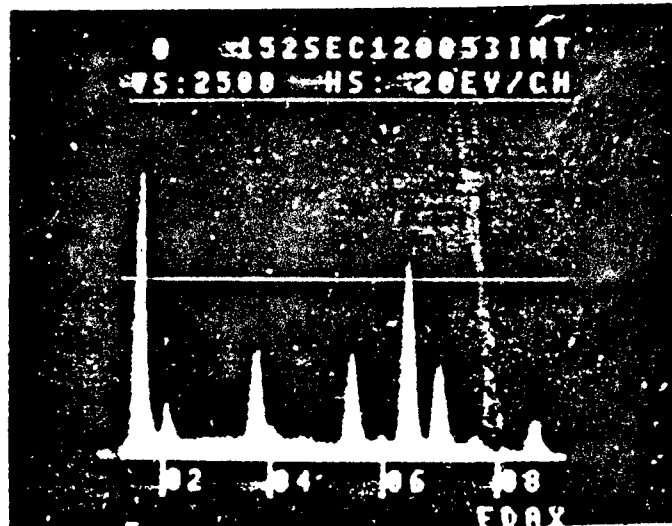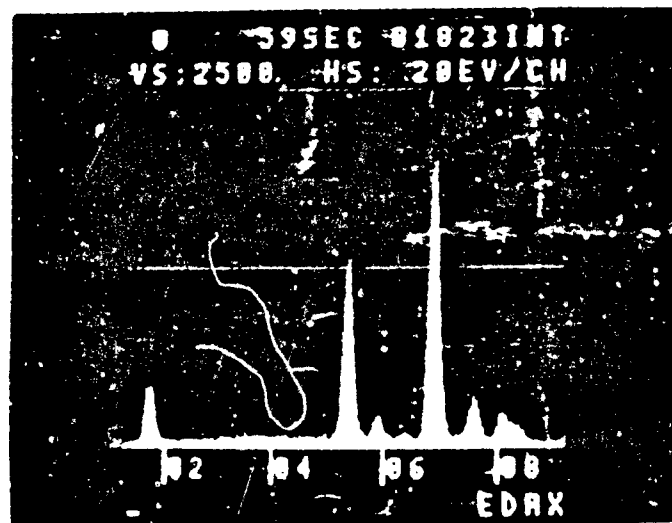
Orange Deposit

Bulk

Figure 9. EDXA spectra of TF-30 turbine blade.

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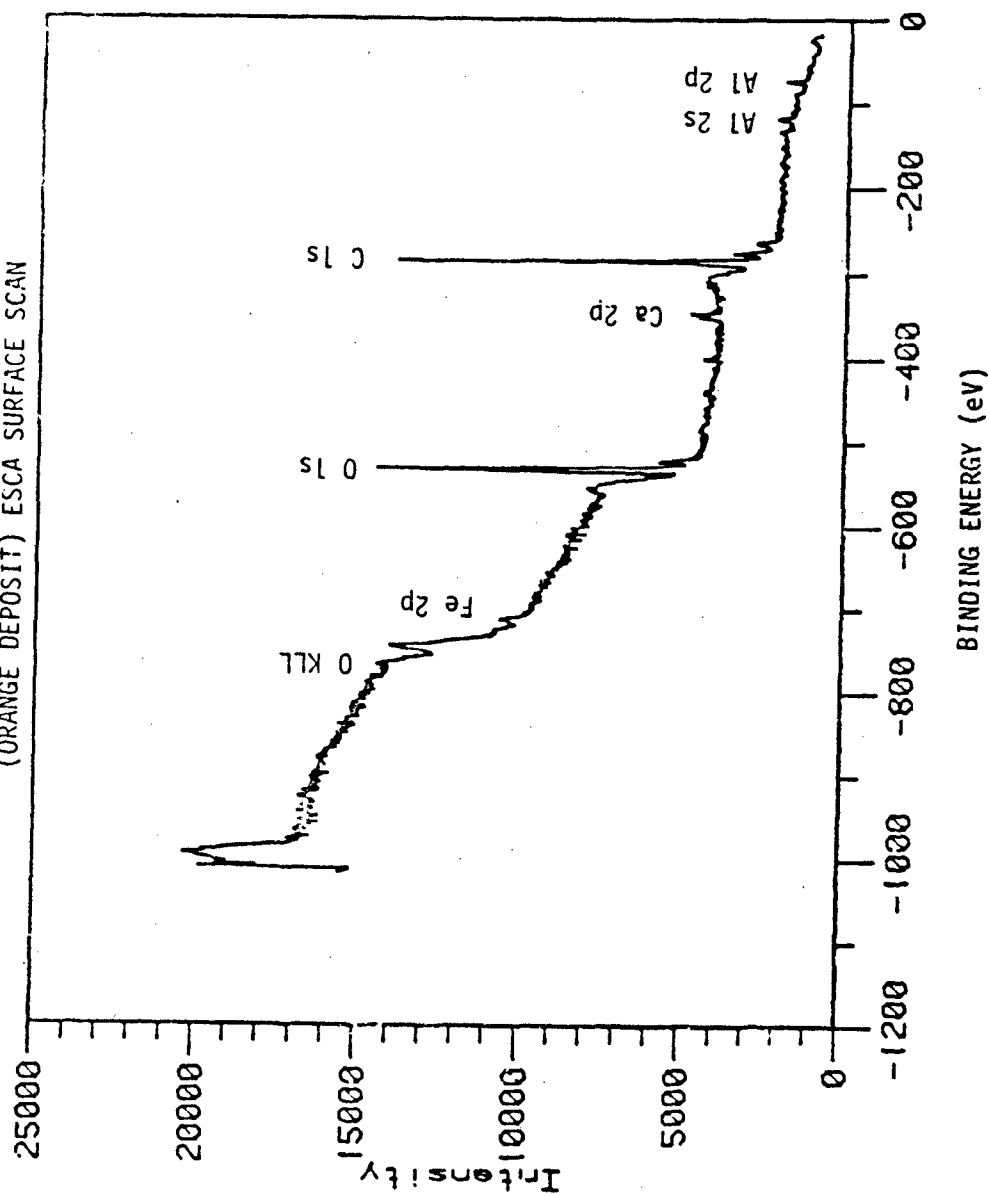
Figure 10. ESCA scan of surface deposit.
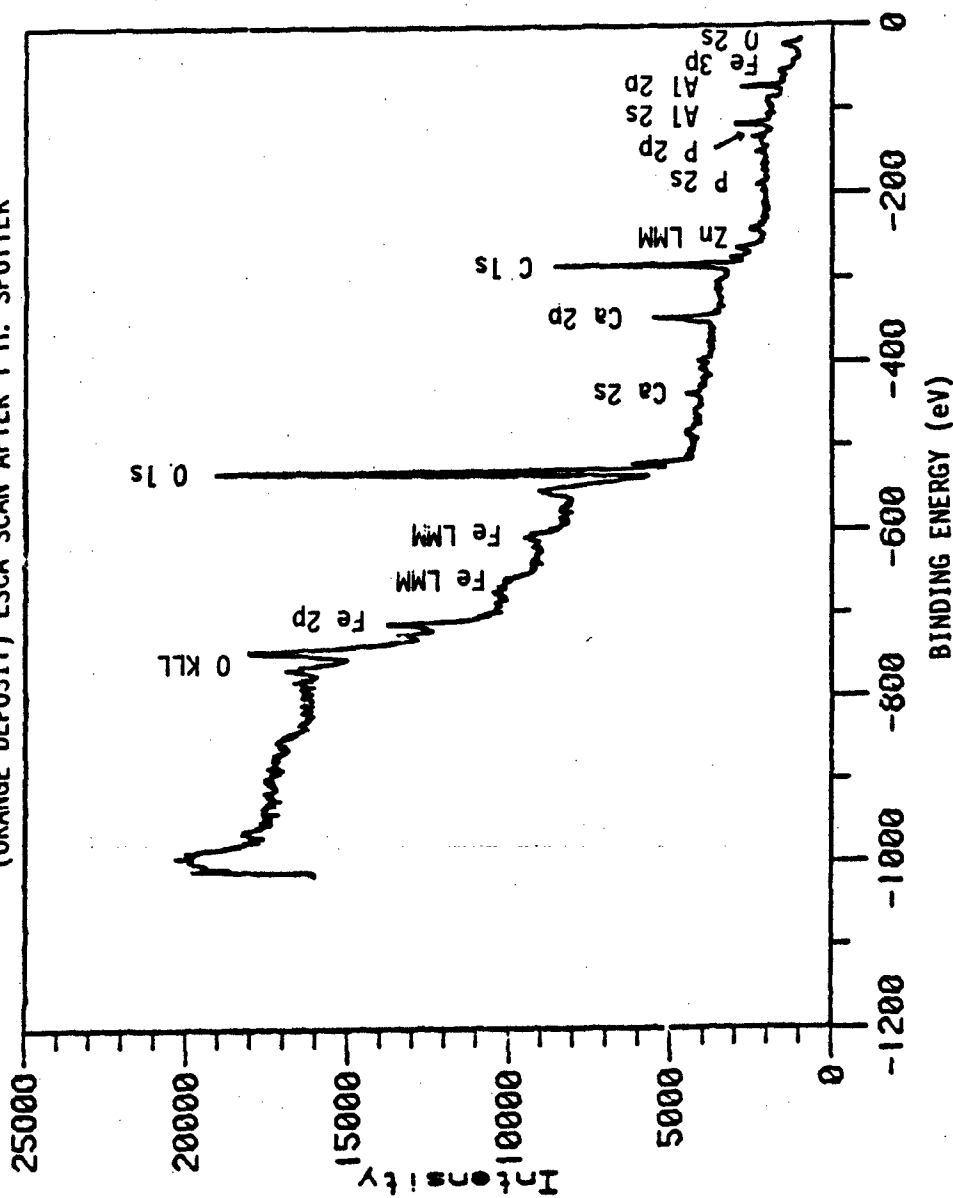
Figure 11. ESCA scan of surface deposit after sputtering.

In summary, analyses were made on a substantial deposit that had formed on a turbine blade during engine testing. Several constituents were found in the deposit that were not present in the alloy itself. The orange coloration of the deposit was probably largely due to the presence of iron oxide.

2. IDENTIFICATION OF NITROGEN CONTAMINANTS IN JP-4 FUEL AND RELATED SAMPLES

JP-4 fuel and related samples were examined for possible contamination with fertilizer solutions during pipeline delivery. The samples submitted were identified as follows:

> 84-POSF-1925 Water Bottoms
> 84-POSF-1926 JP-4 Fuel Sample
> 84-POSF-1927 Filter Element

If there were pipeline contamination with nitrogen solutions, the likely contaminants would be ammonium nitrate and/or urea. Therefore, the investigation was directed toward analyzing for these compounds.

The samples, as submitted, required some preparative treatment prior to carrying out analyses for the compounds of interest. The water bottoms sample was used as received; the fuel sample was extracted with water, then the water extract was analyzed; and the filter element was soaked in water for an extended period of time, and the water then analyzed. Results obtained on these samples are shown in Table 46.

Conclusions

The results obtained with the water bottoms sample (84-POSF-1925), and the filter element (84-POSF-1926) showed rather conclusively that some nitrogen solution contamination had occurred. The contamination is very evident when compared with an AFWAL/POSF tank bottoms sample that was analyzed as a reference sample.

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TABLE 46. ANALYSES FOR NITROGEN CONTAMINANTS

Sample Identification	Total nitrogen[a] mg/L	Nitrate (NO_3-)[b] mg/L	Urea (NH_2CONH_2)[c] mg/L
84-POSF-1925	880	96	86
Water[d] from soaking filter element 84-POSF-1927	306	105	63
Water[e] from extraction of fuel sample 84-POSF-1926	1.5	<.02	-
Water bottoms from AFWAL/POSF storage tank 82-POSF-0541	17	0.34	3

[a]Measured by Howard Laboratories using Kjeldahl method.

[b]Measured by Howard Laboratories using cadmium reduction method.

[c]Measured by Monsanto using Sohio Chemical Test Method SCC-65-8.

[d]3,500 mls. of water for a soak period of 72 hours.

[e]100 mls. of water and 150 mls of fuel - mechanical shaker for 2 hours.

3. IDENTIFICATION OF FUEL FILTER CONTAMINANTS

Contaminants taken from an on-board aircraft fuel filter, sample No. 84-POSF-1950, were submitted for analysis. The sample was observed to be nonhomogeneous, and contained metallic and non-metallic components.

Analyses carried out on this sample utilized two laboratory techniques: (1) Energy Dispersive X-ray Analysis (EDXA), and (2) Inductively Coupled Argon Plasma Spectroscopy (ICP). The EDXA is a nondestructive test where the sample is bombarded by high energy x-rays. As the depth of penetration is small, it is essentially a surface analysis technique. The ICP, on the other hand, is a bulk analysis technique where the sample is dissolved in appropriate solvents before the analysis can be made.

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The sample was difficult to handle as it was so heterogeneous.
Several categories of materials were observed in the sample, e.g.,
aluminum-like, metallic (magnetic), rubber-like, and fibrous.
Physical separation of the sample into the various categories
was carried out before the analyses were undertaken. Results of
the analyses are shown in Table 47.

Iron, aluminum, and calcium were found to be the major components
in the various categories. Other elements were found as shown in
Table 47. Results from the separated fractions appear reasonable.
For example, the calcium value of 11% in the rubber-like fraction
is probably there as calcium carbonate, which is a common binder
material in the rubber industry. The metallic and aluminum-like
particles, as expected, showed the major components to be iron and
aluminum, respectively. Both the rubber-like and fibrous fractions
exhibited a high level of nonmetallic components. The quantitative
results given for the ICP analysis are accurate for that particular
fraction, and should give a good indication of the concentration
of those metals in the original sample.

TABLE 47. ANALYSES OF VARIOUS CATEGORIES

	EDXA[a]	ICP[b]
Aluminum-like	Al, Cr, Ca, Fe	Al[76%], Cu[3%], Fe[3%], Mg[1%]
Metallic (magnetic)	Fe, Cr, Ca, Ni, Zn	Fe[61%], Cr[10%], Ni[5%], Zn[1%]
Rubber-like	Ca, S, Mn, Ti, Cd	Ca[11%], Mn[3%]
Fibrous	Ca, S, Mn, Fe, Ti	Ca[6%], Al[3%], Mn[2%]

[a]The major element found is on the left. The other elements listed are in
decreasing order--going from left to right.

[b]Values are in weight %.

4. IDENTIFICATION OF VENTILATION CONTAMINATION DURING COMBUSTION TESTING

A liquid sample having a cloudy, milky appearance was submitted for analysis. The unknown liquid had been produced during simulated combustion testing. Analytical techniques used in assessing this sample were: (1) Nuclear Magnetic Resonance (NMR), (2) Infrared Spectrophotometry (IR), (3) Energy Dispersive X-Ray Analysis (EDXA) and (4) density measurements.

Results of NMR analyses are shown in Figures 12 and 13. Different solvents were used in the two analyses. In Figure 12, results are with deuterated water as the solvent; in Figure 13, with deuterated dimethyl sulfoxide. From the position of the resonance peak, one can infer large amounts of water present -- no hydrocarbons are seen.

Comparisons of IR spectra of the sample (VC-1) and of tap water are shown in Figures 14 and 15. The spectra are very similar -- the only real difference being an unidentified absorption band on the sample at ~7μ wavelength which was not seen with the water sample.

Density measurements made on VC-1 agreed with the NMR and IR results that the sample was essentially all water. Density at 77°F was 1.0005.

It was thought that titanium dioxide might be present in the liquid sample. Accordingly, the sample was analyzed by EDXA to determine if that were the case. Results, which are given in Figure 16, showed zinc to be the major constituent along with chlorine. No evidence of any titanium was found.
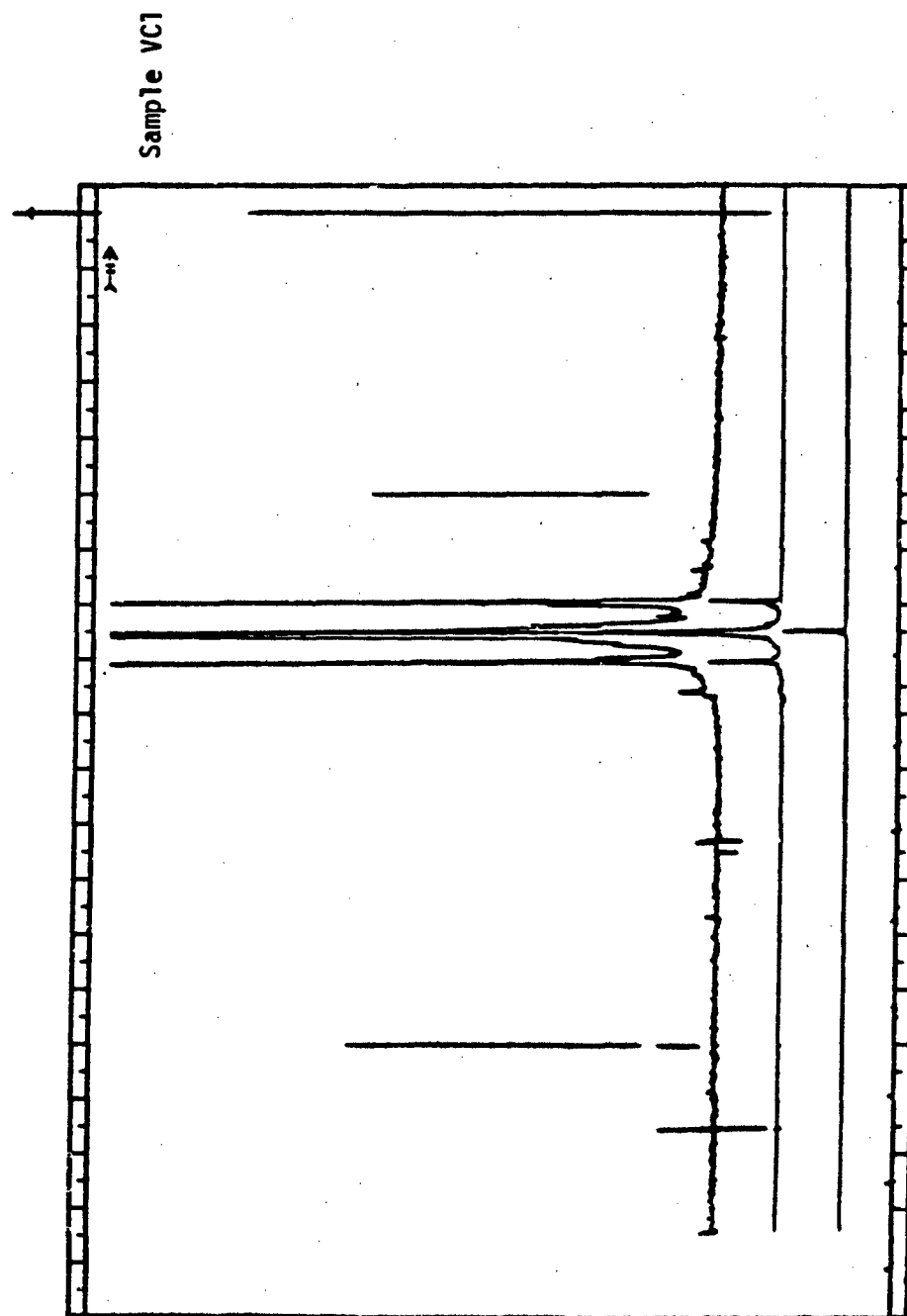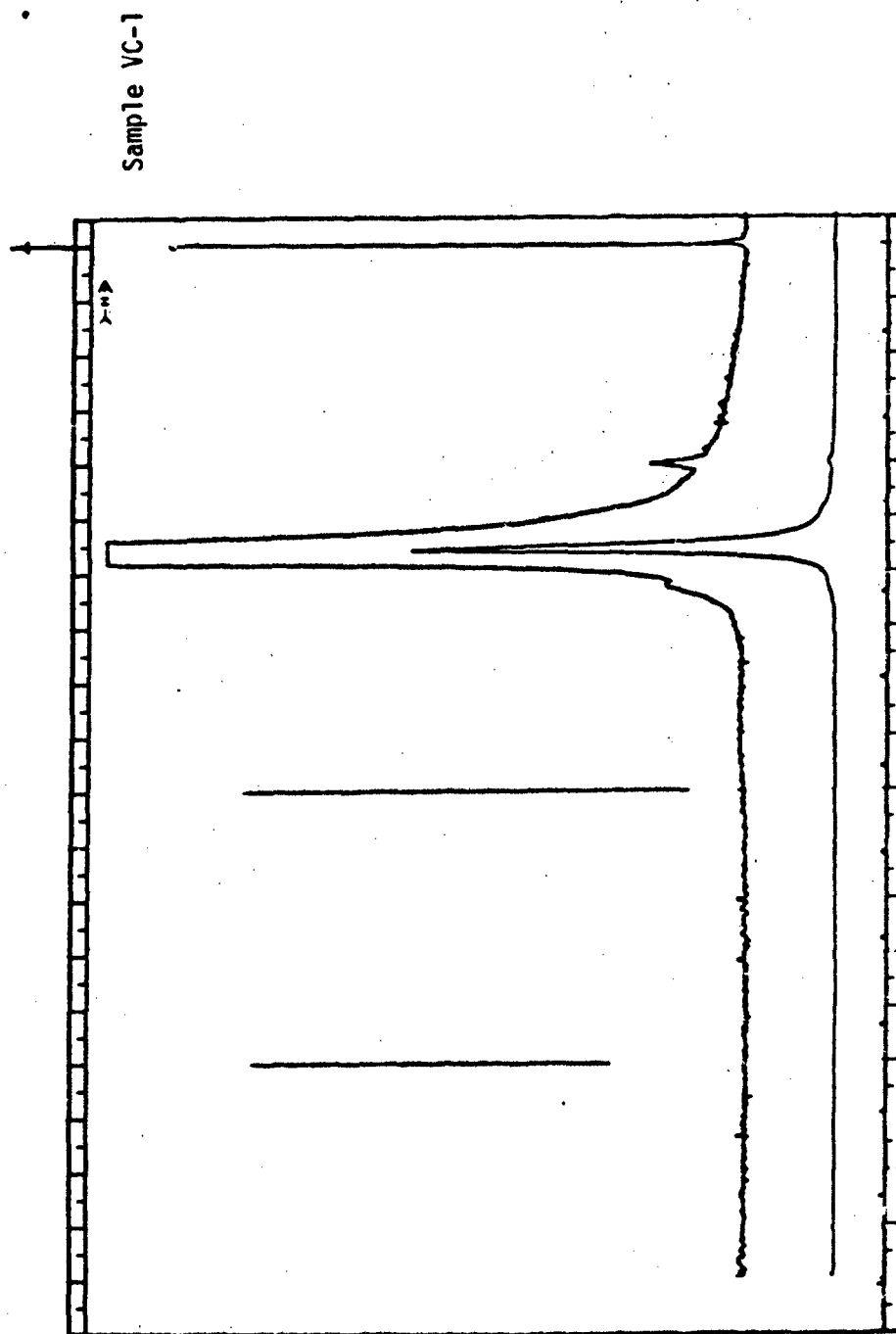
Figure 12. NMR scan of sample VC-1 (D_2O solvent).

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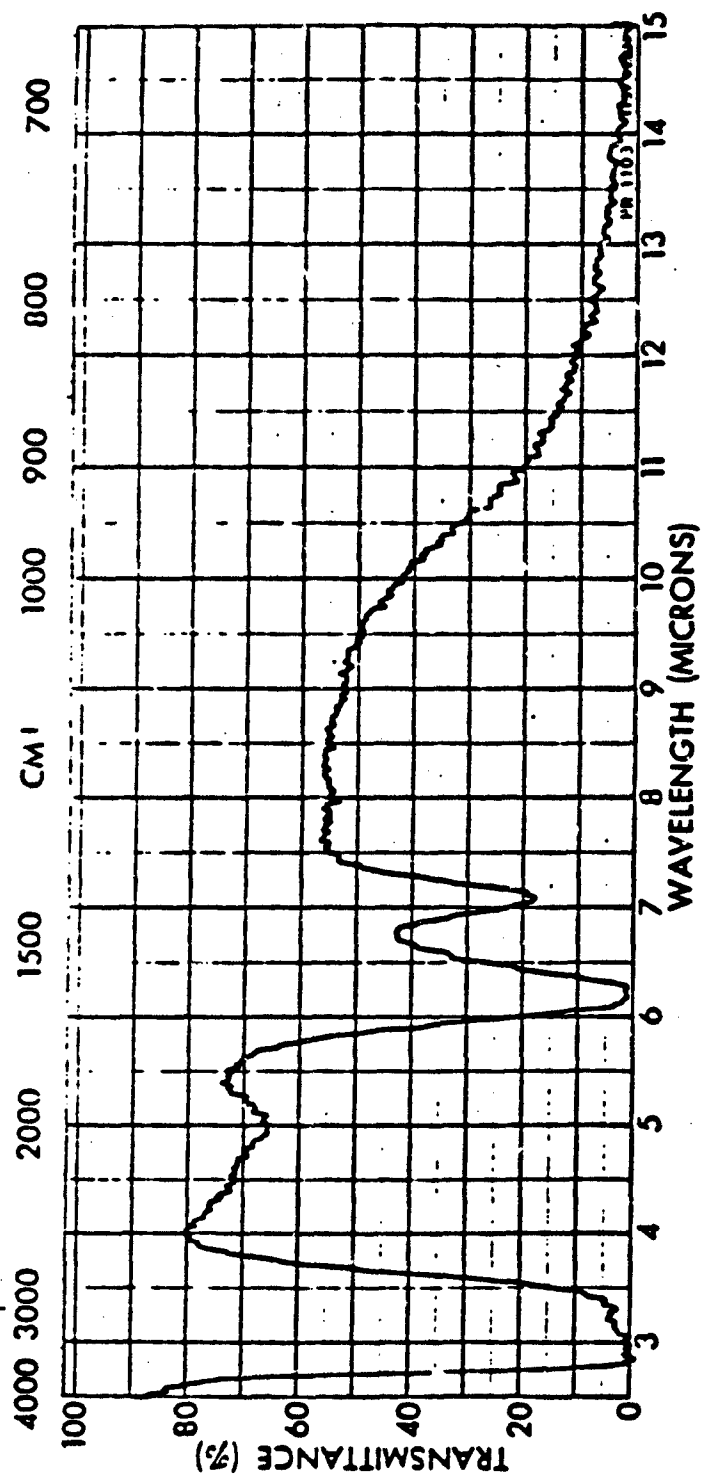Figure 13. NMR scan of Sample VC-1 (deuterated DMSO solvent)

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Figure 14.   IR spectra of VC-1
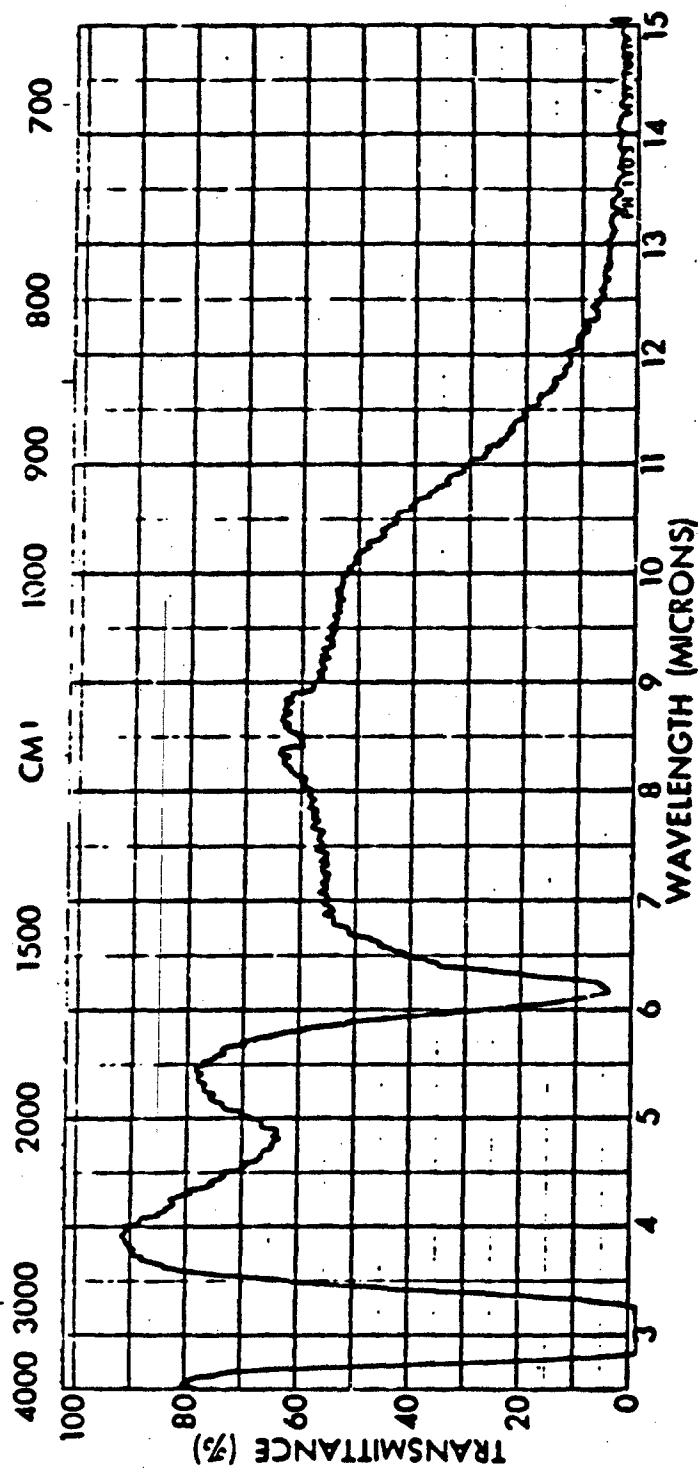
Figure 15.   IR spectra of tap water

```
*  ENTR  MEM:  C  #VC-1  ER = 9
*  SMTH  MEM:  A
*  SMTH  MEM:  C
*  LIST  MEM:  C  VC-1
```

	VC-1		
.570	475.	#	
.571	1814.	#	
1.400	9.	#	
1.795	4257.	#	N, mα
2.055	1010.	#	
2.232	979.	#	
2.627	61145.	#	Cl, Kα
3.055	101.	#	
3.252	90.		
3.654	235.	#	C , Kα
3.910	14.		
4.455	38.		
5.960	39.		
6.406	221.	#	Fe, Kα
6.875	324.		
7.757	267.	#	
7.938	1157.	#	
8.108	3369.	#	
8.283	4352.	#	
8.619	266120.	#	
9.030	1275.	#	Zn, Kα
9.217	2035.	#	
9.561	43020.	#	Zn, Kβ
9.920	520.	#	
10.490	26.		
11.250	175.	#	
12.130	28.		
12.650	68.	#	
12.930	6.		
13.830	20.	#	
14.100	1.	#	
14.140	1.	#	
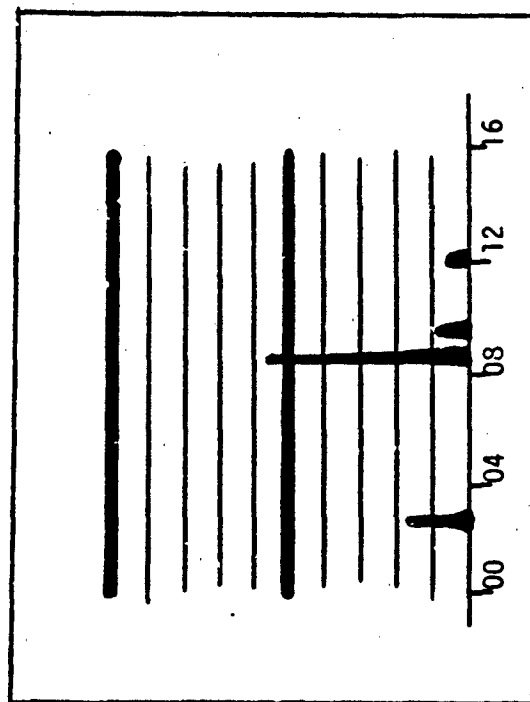
Figure 16.   EDXA spectra of VC-1

Conclusions

Sample VC-1 is largely water. Other constituents found were zinc and chlorine. The sample was acidic (pH of 4). The presence of zinc could be explained by HCl washing over a galvanized surface.

5. LEAD CONTENT IN FUELS FROM F100 PROGRAM

Six samples of JP-4 aviation fuel from the F100 program were submitted for lead analysis. The purpose of this work was to determine trace levels of lead which could be attributed to tetraethyl lead (TEL) contamination in the fuel handling system.

Results were obtained using atomic absorption spectrometry (AA). Acid extraction of lead from the fuel samples was carried out using ultrapure nitric acid, Ultrex®. (Initial attempts at using hydrochloric acid were not successful). The acid extracts were diluted with distilled water in such a fashion that the acid-water extract contained a concentration of lead that was 10 times that in the fuel. The acid-water extracts were then analyzed by direct aspiration. Results of the analyses are given in Table 48.

As shown in Table 48, a reference lead standard was prepared using dilute TEL in isooctane. Analysis of the standard was excellent, as the result obtained was ~97% of the theoretical value.

The results in Table 48 show a wide range of lead levels in the JP-4 fuels. Sample 83-POSF-1157 was by far the highest, having 1235 ppb of lead. This contrasted sharply with 83-POSF-1084 which had no detectible lead.

It is of interest to note the problems encountered in the analysis itself. Extraction of the Pb from the fuel with HCl was

unsuccessful as only a partial extraction was achieved. Whether this was due to the inability of the HCl to break the lead-ethyl bond, or the limited solubility of $PbCl_2$ in water, the HCl didn't do the job. Because of this, a decision was made to try a nitric acid extraction. Results were excellent, and the quantitative extraction and transfer of the lead from the fuel to the acid-water was achieved.

There was some thought that the high lead levels might be partially due to the fuel contacting solder (a lead-tin alloy in the storage cans). In order to check this out, inductively coupled plasma spectroscopy (ICP) was used to specifically analyze for tin on samples 83-POSF-1157 and 83-POSF-1006. In both cases no tin was detected. It seems certain that the lead seen in these fuels has come from TEL.

TABLE 48. LEAD CONTENT IN JP-4 FUELS

Sample I.D.	Pb	
	µg/mL	ppb[d]
83-POSF-1006	0.437	583
83-POSF-1084	N.D.[a]	N.D.
83-POSF-1086	0.141	188
83-POSF-1157	0.926	1235
83-POSF-1157[b]	0.032	43
83-POSF-1272	0.183	244
83-POSF-1272[c]	0.004	5
83-POSF-1273	0.132	176
Blank	N.D.	N.D.
Isooctane spiked with TEL, 0.449 µg/mL	0.437	633
2nd extraction of spiked isooctane	0.014	20

[a] not detected
[b] 2nd extraction of same sample
[c] 2nd extraction of same sample
[d] a density of 0.75 g/ml was used in calculating the ppb

6. DETERMINATION OF TRACE METALS IN JP-8 FUELS

A group of JP-8 fuels had produced a blue deposit during JFTOT
testing at 260°C. Samples were prepared for analysis of copper,
zinc, and lead by inductively coupled plasma spectroscopy (ICP).
Extraction of the fuel samples was carried out with high-purity
hydrochloric acid, and then the extract diluted with distilled,
deionized water. Selection of volumes was such that the resultant
acid-water extract contained a concentration of the metals that
was 20 times that in the fuels. The acid-water extracts were then
analyzed by direct aspiration using a 1SA Model JY48P ICP spectrom-
eter. Results of the analyses are given in Table 49.

Conclusion

The level of copper in the JP-8 fuels is sufficient to cause the
blue deposits found in the JFTOT tester.

TABLE 49. TRACE METALS IN JP-8 SAMPLES BY ICP SPECTROMETRY

Elements of detection:	Cu	Zn	Pb
Instrument quantification limit, LOQ, ppb:	67	170	74
LOQ after concentration[a], ppb:	3.4	8.5	3.7
Acid extraction blank[b], ppb:	X	X	X
Concentration of elements in fuel samples[c], ppb			
84-POSF-1891	47	X	X
84-POSF-1897	21	X	X
84-POSF-1900	14	X	X
84-POSF-1902	25	X	X
84-POSF-1903	23	X	X

[a] Fuel samples were effectively concentrated by a factor of 20
during the acid extraction.

[b] Aqueous ultrapure acid was used to extract the fuel samples. An
"X" shows that the element was not detected at the instrument LOQ.

[c] An "X" shows that the element was not detected at the LOQ after
concentration. The numerical values were obtained by dividing the
observed value by 20 to take into account the concentration effect.

Another group of JP-8 fuels had produced blue and brown deposits during JFTOT testing at 260°C. Samples were prepared for metals analysis by inductively coupled plasma spectroscopy (ICP). Extraction of the fuel samples was carried out with Ultrex® high-purity nitric acid, and then the extract was diluted with distilled, deionized water. Selection of volumes was such that the resultant acid-water extract contained a concentration of the metals that was 10 times that in the fuel. The acid-water extracts were then analyzed by direct aspiration using a 1SA Model JY48P ICP spectrometer. Results of the analyses are given in Table 50.

Conclusion

The level of copper in the JP-8 fuels is quite high, and probably explains the blue deposits found in the JFTOT tester. Other metals detected were Ca, Mg, and Ba. No Pb or Sn was detected.

TABLE 50. TRACE METALS IN JP-8 SAMPLES BY ICP SPECTROMETRY

Elements of Detection	Cu	Ca	Mg	Ba
Instrument quantification limit, LOQ, ppb:	23	33	5	4
LOQ after concentration[a], ppb:	2.3	3.3	0.5	0.4
Acid extraction blank[b], ppb:	X	X	X	X
Concentration of elements in fuel samples, ppb				
84-POSF-2076	165	26	14	3
84-POSF-2077	75	34	16	9

[a]Fuel samples were effectively concentrated by a factor of 10 during the acid extraction.
[b]Aqueous ultrapure acid was used to extract the fuel samples. An "X" shows that the element was not detected at the instrument LOQ.

7. ANALYSIS OF A DEPOSIT FOUND IN A SHALE JP-4 FUEL SAMPLE

A deposit was found in a 5-gallon can of shale JP-4 jet fuel, and submitted for analysis (Sample No. 83-POSF-1487). The deposit was light gray in color and had a rubbery consistency. The investigative effort on this sample was restricted because of our inability to find a solvent that would dissolve the material. A list of the solvents tried without success is as follows: water, methanol, ethanol, chloroform, acetone, dimethyl sulfoxide, 0.1N. sodium hydroxide, and 0.1N hydrochloric acid.

An initial examination of the sample was made using X-ray fluorescence spectrometry, EDAX International Model 707A. This qualitative analytical technique showed calcium to be a major constituent with traces of potassium, iron, sulfur, and chlorine also being present.

Another nondestructive test whereby the sample was examined in its solid form was infrared attenuated total reflectance (ATR). Accordingly, this test was carried out using a Connecticut Instrument Corporation ATR attachment on a Perkin-Elmer Model 137 infrared spectrophotometer. Results are graphically displayed in Figure 17. The spectra contained in Figure 17 show the deposit material with a teflon backing, Spectrum 1, and the teflon backing itself, Spectrum 2. In examining Spectrum 1, bands can be seen which are characteristic of calcium carbonate -- strong band at about 7 μ, and peaks at 11.5 and 14.4 μ (2). Other reflectance bands are probably caused by the organic constituents of this deposit.

Conclusions

Analytical results strongly point to this deposit containing calcium carbonate as a major component. Further, calcium carbonate is widely used as a rubber filler, and the deposit material is

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certainly rubbery, and impervious to a wide range of solvents. It seems likely that this deposit is a fuel tank sealant, or some other similar elastomeric material.
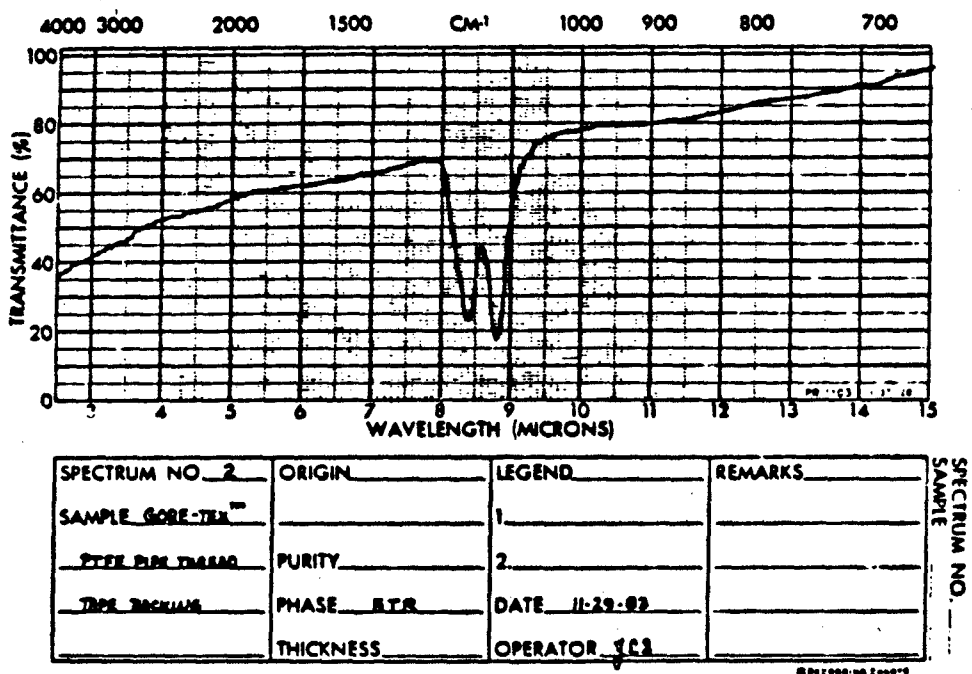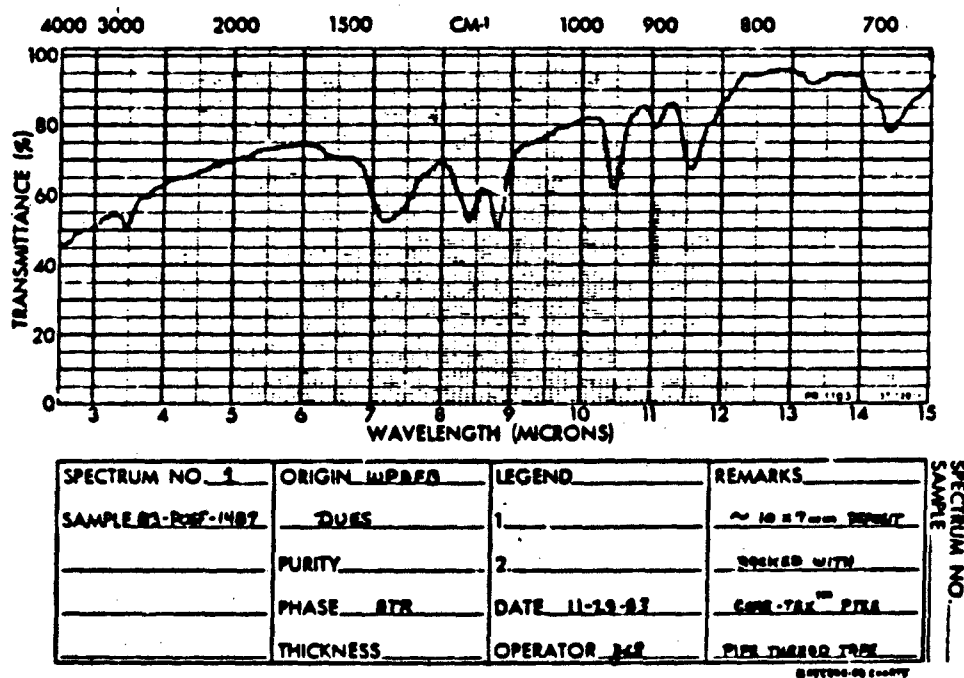
Figure 17. ATR infrared spectra of deposit and backing material.

8. <u>TRACE METALS ANALYSIS OF SHALE JP-4 FUELS</u>

Four samples of shale JP-4 fuel were submitted for trace metals analysis. The fuel under study was being used in a very important TF30 AMT engine test as WPAFB. The test had been interrupted when a high response of an "ion probe" was encountered. Subsequently, samples of fuel were taken from the tanks as well as engine inlet samples. The samples were identified as follows:

83-POSF-1601	Tank B-3
83-POSF-1667	Tank B-2
83-POSF-1704	Cycle #210
83-POSF-1705	Cycle #224

Samples were prepared for analysis of Al, Cr, Cu, Fe, Mg, and Zn by inductively coupled spectrometry (ICP). Extraction of the fuel samples was carried out with high-purity hydrochloric acid, and then diluted with distilled, deionized water. Selection of volumes was such that the resultant acid-water extract contained a concentration of the metals that was 14 times that in the fuels. The acid-water extracts were then analyzed by direct aspiration using a 1SA Model HY48P ICP spectrometer. Results of the analyses are shown in Table 51.

Conclusion

The level of trace metals in the submitted samples was very low. Therefore, it is extremely unlikely that the "ion probe" upset was caused by any problem with the fuel.

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TABLE 51. DETERMINATION OF TRACE METALS IN SHALE
JP-4 SAMPLES BY ICP SPECTROMETRY

Elements of detection:	Al	Cr	Cu	Fe	Mg	Zn
Instrument quantification limit, LOQ, ppb:	200	6	15	5	5	2
LOQ after concentration,[a] ppb:	14	0.4	1	0.4	0.4	0.1
Acid extraction blank,[b] ppb:	X	X	X	1	0.5	2
Concentration of elements[c] in fuel samples, ppb						
83-POSF-1601	X	X	X	16	1	4
83-POSF-1667	X	1	3	21	1	3
83-POSF-1704	X	X	3	11	1	2
83-POSF-1705	X	X	2	12	1	4
Analyses of reference standards						
ERA 8701 % recovered	97.5	93.4	100.0	95.8	–	103.0
ERA 9401 % recovered	101.0	97.2	99.1	94.1	–	99.7

[a]Fuel samples were effectively concentrated by a factor of 14 during the acid extraction.

[b]Aqueous ultrapure acid was used to extract the fuel samples. An "X" shows that the element was not detected as the instrument LOQ.

[c]An "X" shows that the element was not detected as the LOQ after concentration. The numerical values were obtained by dividing the observed value by 14 to take into account the concentration effect, then subtracting any blank value obtained. These values represent the element concentration in the original fuels.

SECTION IV

SPECIAL PROJECTS AND INVESTIGATIONS

Special projects and investigations were conducted to aid in improving existing test methodology, make measurements of a non-routine nature, or aid in the solution of operational problems.

1. ## EVALUATION OF METHODS FOR RAPID AND ACCURATE
   ## DETERMINATION OF SULFUR

A number of methods for the determination of sulfur and nitrogen in hydrocarbon fuels have been investigated for possible Air Force use in evaluating hydroprocessed fuels. Criteria for judging suitability include accuracy and precision, limits of detection, ease of operation, reliability, expected turnaround time, and capital and operating costs.

Summary of Approaches for Sulfur Analysis

Many methods which have been successfully used for sulfur analysis include, as a preliminary step, the oxidative combustion of the sample followed by collection of sulfur oxides. Quantitative measurement is then made by any one of a variety of techniques. Leco Corporation's Model 765-100 digital titrator [3,4] for example, is an integral part of the Leco carbon/sulfur analyzer. In this system, combustion takes place in an oxygen stream which passes over the sample in an induction furnace. Liberated sulfur dioxide is automatically titrated with iodine.

Ion chromatography [4] is commonly used for the measurement of sulfate ions resulting from the oxidation of the sample in a Parr oxygen bomb containing a carbonate/bicarbonate absorbing solution.

Ion chromatography provides both good accuracy and high sensitivity. The bomb combustion technique, however, is rather labor intensive.

An important method which was used widely in the petroleum industry at one time is the Wickbold method (ASTM D2785-70). This method involves burning the sample in an oxy-hydrogen atomizer burner. The oxides of sulfur are absorbed and oxidized to sulfuric acid by a hydrogen peroxide solution. The sulfate ions are then determined by either a barium perchlorate titration or by turbimetry. Though quite sensitive (LDL, 2 ppm), the method requires long burning times for analysis of sulfur at low levels. The Wickbold method now has been largely replaced by other more precise and less time consuming methods.

Many of the current procedures still use oxidative combustion as a preliminary step. Microcoulometry [5], for example, has been a popular technique for sulfur analysis (ASTM D 3120) in the petroleum industry. The Dohrman microcoulometer is an instrument in which the total sample is combusted, with oxidation products containing SO_2 being swept into a titration cell. In the cell, the analyte ions are consumed by titrant, the latter being continuously electrolytically replaced. The coulometric current required for this process is proportional to the concentration of the analyte.

Success in using the technique depends upon controlling combustion conditions and rates, so that complete oxidation takes place. A "Constant Rate Injector," CRI, is now available from Dohrmann Laboratories to control sample injection. A recent publication [6] coauthored by investigators at Sun Oil Company (Tulsa) and R. T. Moore of Dohrmann, describes the CRI accessory as well as a granular tin scrubber used to eliminate negative interferences due to nitrogen, chlorine, and peroxides. In a telephone conversation, the Sun Oil Company authors expressed the belief that the

system now overcomes past difficulties associated with sulfur analysis by microcoulometry. Sun uses the technique almost exclusively for analysis of relatively clean oils in which sulfur is present only in the part-per-million range.

Monsanto's physical sciences center in St. Louis, as well as other Monsanto laboratories, use microcoulometry for sulfur analysis. Monsanto analysts report good results much of the time but have experienced difficulties if sulfur concentrations in the sample vary over a wide range, especially when concentration orders of magnitude are not readily predictable. A poor combustion or sample overloading has been reported to occasionally put the instrument out of service for a short though frustrating period of time. More recent improvements in microcoulometer methodology are the adaption of a boat inlet and development of a high capacity combustion tube. These modifications have resulted in better analyses for sulfur in petroleum distillates. The methodology is under ininvestigation by ASTM Subcommittee D-16 and is presently undergoing round-robin testing for acceptance into Method ASTM D 1552 for analysis of sulfur in hydrocarbons boiling above 177°C and containing not less than 0.06% sulfur.

A number of other analytical techniques have been used for the determination of sulfur in petroleum products. Flameless atomic absorption [7] has been used to measure sulfur dioxide from combustion of the sample after trapping in tetrachloromercurate then releasing through a quartz-windowed flow-through cell for measurement of absorption at 210 nm.

The Houston-Atlas sulfur analyzer uses an approach that is quite different from those discussed previously. It consists of a hydrogenation unit that converts all sulfur in the sample to hydrogen sulfide (H_2S), which is then determined with a unique H_2S analyzer. The sample is injected at a constant rate into a

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flowing hydrogen stream in the hydrogenation apparatus. The sample and hydrogen are pyrolyzed, at a temperature of about 1300°C or above, to convert sulfur compounds to hydrogen sulfide. In the readout unit, the H_2S flows into the reaction window of the sample chamber where it passes over a lead acetate impregnated sensing tape, causing its surface to darken from lead sulfide formation. The rate of darkening is proportional to the H_2S concentration, or to the concentration of sulfur in the sample. This sensing method has been known to be accurate, reproducible, and virtually free of interference.

Another type of analytical technique which has been used for sulfur analysis is x-ray fluorescence. In this technique the sample is placed in an x-ray beam where spectral lines of the elements present are excited. Elements having atomic numbers of 13 or greater exhibit spectral lines of sufficient energy for detection and measurement. The sulfur $K\alpha$ line can, thus, be excited and measured giving sulfur quantitation without destroying or changing the sample.

X-ray fluorescence instruments may disperse or separate the emission lines of the various elements in one of several ways. A crystal monochromator, or analyzing crystal, may be used to separate each line according to wavelength using the principle of diffraction, while very good resolution of lines is obtained in this way, only a part of the fluorescent radiation reaches the detector. Alternatively, the lines may be separated electronically on the basis of their energies as in the energy dispersive instruments. Separation is not as good but a much higher percentage of the total fluorescence radiation is detected. Thus sensitivity is greater. Finally, if it is known that one or a limited number of elements that can be excited to x-ray emission are present in the sample, a non-dispersive instrument may be used. In this instrument, the only separation that takes place is accomplished by filtration.

Thus a discrete "window" is obtained for the detection of a specific spectral line for the single element to be determined.

ASTM Method D 2622-77, "Sulfur in Petroleum Products (x-ray spectrographic method)" requires an instrument having an analyzing crystal monochromator. Other methods for sulfur have been developed using energy dispersive instruments. Recently, Universal Oil Products, Inc. which provides an analytical method subscription service, released UOP Method 836-82, "Sulfur in Distillate and Residual Oils by X-ray Fluorescence." The method employs a non-dispersive XRF unit and specifically mentions the Horiba Model SLFA-800 non-dispersive XRF sulfur analyzer.

Specific Techniques Considered

Three instruments for sulfur analysis, offering unique features and characteristics have been selected for further consideration. These are the microcoulometer, the hydrogenation/H_2 analyzer, and x-ray fluorescence spectrometer.

1. The Dohrman Microcoulometer

Microcoulometry has the advantage of having been in use in the petroleum industry for some time and being included in ASTM methods. It is used rather widely, especially for very low levels of sulfur and for routine samples of similar nature. This instrument has been used in several Monsanto locations though in one location it has now been replaced by a Houston-Atlas analyzer (described below). It is particularly useful for very low levels of sulfur, i.e., sub-ppm to 100 ppm.

2. Houston-Atlas Sulfur Analyzer

This analytical system, consisting of a Model 856 hydrogenator and Model 825 hydrogen sulfide analyzer is sufficiently simple to

permit operation by non-technical personnel. The pyrolysis approach used in the hydrogenator should not be confused with the oxidative combustion used in many analyzers. The process is very quantitive and is not subject to interferences. Discussions with Monsanto analysts who use the Houston-Atlas system indicate a high level of satisfaction with the unit. Responding to a request for candid comments on the system's reliability, only one area where failure had occurred was reported. In the unit located in Monsanto's Chocolate Bayou facility (Texas), the furnace elements failed after 4 to 5 month's usage; they were quickly replaced by Houston-Atlas at no cost.

3. X-ray Fluorescence

As mentioned in the summary of techniques, three types of XRF instruments are available, the crystal monochromator type, the energy dispersive type, and the non-dispersive type. ASTM Method D 2622-77 requires the crystal monochromator instrument. This unit, which includes a precision goniometer, typically sells for a price in excess of $50,000. This cost precludes its further consideration based on the price criterion. The energy dispersive system, too, is substantially more than the $20,000 target price--perhaps twice that amount depending on model and accessories purchased.

One XRF instrument which has been specifically manufactured for sulfur analysis is the model SLFA-800 non-dispersive analyzer by Horiba Instruments, Inc. In all x-ray fluorescence measurements the effect of the sample matrix on results must be considered. The intensity of an XRF line may vary considerably for the same concentration of the element giving rise to it, depending on the composition of the bulk of the sample (matrix). In the Horiba instrument, this effect is minimized by an automatic C/H ratio compensation circuit. The fluorescent radiation from the samples is filtered to remove radiation originating from elements other than sulfur. The accuracy of the instrument depends upon

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the extent that these operations are successfully carried out.
High levels of metals, particularly lead and molybdenum at con-
centrations above 100 ppm interfere with the analysis.

The XRF techniques, and specifically the Horiba SLFA-800 have a
number of attractive characteristics. It provides rapid, reproduc-
ible results with virtually no sample preparation. The method is
useful over the range of 100 ppm to 5 weight percent sulfur.
Samples of higher concentrations can, of course, be diluted.

Comparison of Instruments

For convenience the three instruments selected for consideration
are referred to by letter designations as:

    A. Dohrmann Microcoulometer
    B. Houston-Atlas Sulfur Analyzer
    C. Horiba SLFA-800 XRF

1. Price
    A. $18,600
    B. $22,000
    C. $18,000

2. Detection Limit
    A. Fractional parts per million
    B. Fractional parts per million
    C. 100 ppm

3. Standard methods available
    A. ASTM D 3120
    B. ASTM D 4045
    C. UOP 836-82

4. Repeatibility
   A. Differences from average no greater than 28% at the 95% confidence level in the 3-100 ppm concentration range
   B. 16% to 1.6% over the 1 to 100 ppm range
   C. 5.6% to 1.8% over the 0.02 to 4.5% range

5. Analysis time
   A. 5-7 min
   B. 5-7 min
   C. 5-7 min

6. Supplies required
   A. High purity (99.995%) nitrogen, helium or argon
   B. Hydrogen and nitrogen compressed gas; lead acetate sensing tape (100 ft reel, 4,200 sples.)
   C. Disposable sample holders

7. Interference
   A. Halide at concentrations greater than 10 times the sulfur level; total heavy metal concentration (e.g., Ni, V, Pb) in excess of 500 ppm
   B. None known
   C. Lead and molybdenum at concentration in excess of 100 ppm, high level of other metals

Conclusions

Conclusions are based on scientific publications, sales literature, personal discussions and ASTM/UOP methodology. Two instruments, the Houston-Atlas analyzer and the Horiba Model SFLA-800 x-ray fluorescence analyzer, have merit as reliable and reasonably trouble free instruments for sulfur analysis. The Horiba SFLA-800 analyzer is non-destructive, rapid, easy to use, and requires a minimum of supplies.

A major concern regarding its overall accuracy for a wide variety of sample types has been largely eliminated by discussions with

several SLFA-800 users. Chemists at Clark Oil & Refinery Corp.
(Blue Island, IL) and Mobil Oil Co. (Joliet, IL) when contacted
independently, spoke highly of the instrument. Mobil has tested
the instrument on leaded gasoline, comparing the sulfur value with
one obtained by the Lamp Method. The agreement was excellent
despite the known interference shown by lead. Both of the above
laboratories use the instrument for sulfur in the 0.02 to 5%
range. Mobil keeps the instrument in operation 24 hrs. per day
and has owned the instrument for two years with virtually no main-
tenance problems.

The remaining concern regarding the Horiba SLFA-800 is its high
detection limit. Sulfur below 100 parts per million cannot be
detected with the system.

The Houston-Atlas instrument is the instrument of choice if sulfur
in the 1-100 ppm range must be included in the determination.
This instrument is recommended based on its lack of interferences,
low detection limits and generally reliable performance.

The Mobil Oil Company laboratory contacted regarding the Horiba
SLFA-800 plans to purchase a Houston-Atlas analyzer for lower
levels of sulfur. Monsanto laboratories praise the performance
of the instrument. The Houston-Atlas analyzer, because of its
constant rate injection system, can handle high concentrations
as well as fractional parts per million of sulfur. Its price
is about 10% higher than the $20,000 target price established as
a selection criterion.

2. EFFECTS OF FUEL CORROSION INHIBITOR ADDITIVES ON THE ELECTRICAL CONDUCTIVITY OF FUELS CONTAINING ANTISTATIC ADDITIVES

The purpose of this task was to determine the effects of three fuel corrosion inhibitors on the electrical conductivity of 12 fuels (six base fuels; two antistatic additives). The base fuels are identified in Table 52.

TABLE 52. BASE FUELS

Sample No.	Fuel Type
82-POSF-0708	Petroleum JP-4
82-POSF-0883	Petroleum Jet A
82-POSF-0113	Shale JP-4
83-POSF-1330	Petroleum JP-4
83-POSF-1329	Petroleum JP-8
83-POSF-1333	70% Isooctane/30% Toluene

These base fuels were mixed with two antistatic additives at the Aero Propulsion Laboratory with the objective being to obtain 12 fuels whose electrical conductivities were 400± 100 pS/m. Mixing of the fuels was carried out on a paint shaker for at least 30 minutes, and then the fuel was allowed to equilibrate for 24 hours. Conductivity measurements were made, and the process repeated as necessary until proper conductivities were achieved. The fuel-additive compositions are given in Table 53.

The blends listed in Table 53 were delivered to Monsanto in five-gallon cans on 27 February 1984. Also provided were 48 epoxy-lined deactivated one-gallon cans in which to carry out the addition studies with the three corrosion inhibitors, which are listed in Table 54.

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The base fuels in the five-gallon cans were subsequently transferred
in part to deactivated one-gallon cans (and labeled base control),
and other deactivated one-gallon cans where the various additions
of corrosion inhibitors were performed.   In all cases, the maximum

TABLE 53. FUEL BLENDS

	Additive	Concentration of Additive (ppm)	Conductivity* (pS/m)
82-POSF-0708	Stadis 450	1.3	400
82-POSF-0708	ASA-3	1.13	400
82-POSF-0883	Stadis 450	1.6	370
82-POSF-0883	ASA-3	1.4	360
82-POSF-0113	Stadis 450	0.93	430
82-POSF-0113	ASA-3	0.85	400
83-POSF-1330	Stadis 450	1.3	420
83-POSF-1330	ASA-3	1.0	400
83-POSF-1329	Stadis 450	1.46	370
83-POSF-1329	ASA-3	1.4	420
83-POSF-1333	Stadis 450	0.56	350
83-POSF-1333	ASA-3	1.3	430

*Conductivity values were obtained with a hand-held EMCEE Meter, Model 1151,
ID# JH7183

TABLE 54. CORROSION INHIBITORS EVALUATED

Name	Sample Date	Concentration, lbs/1000 bbls.	
		Min. Eff. Conc.	Max. Allowable Conc.
Apollo PRI-19	September 1983	3	8
Hitec E-515	30 September 1983	7.5	16
Lubrizol 541	15 September 1983	3	6

allowable concentration of each corrosion inhibitor was added
first. Mixing was carried out on all samples for a one-hour per-
iod with a mechanical shaker. Equilibration of the samples was
maintained for at least 24 hours before electrical conductivity
measurements were made, according to ASTM D 3114. Where maximum

allowable concentrations of corrosion inhibitors caused electrical conductivity changes greater than ±40% from the control sample, stepwise reductions in concentration were made until either the conductivity change was less than ±40%, or the minimum effective concentration was reached. (In all cases, each new blend was mixed for one hour, and equilibrated for 24 hours, before electrical conductivity measurements were made). A summary of the fuel blends and electrical conductivity readings are given in Table 55.

The original blends containing the antistatic additives were prepared at AFWAL/POSF so as to have electrical conductivities of 400 ± 100 pS/m. Some of the fuels tested at Monsanto did not fall in this range of conductivities. It may have been that some of the fuels were not equilibrated when tested at WPAFB; also there is a definite bias between the Portable Meter Method, ASTM D 2624, and the Precision Conductivity Method, ASTM D 3114. (This will be discussed later in the report).

In examining the data in Table 55, one sees clear evidence of additive interactions, some being fuel-dependent but most not fuel dependent. The findings are summarized in Table 56.

After the extensive testing, compiled in Table 55, was completed, it was decided to compare the two methods of electrical conductivity measurement used in the study. (The preliminary results by AFWAL/POSF personnel used the EMCEE handheld meter, ASTM D 2624; while the Monsanto results in Table 55 used Precision Method, ASTM D 3114). The same meter that had been used at AFWAL/POSF was borrowed for this effort. Electrical conductivity measurements on all base fuels were determined at Monsanto by both methods. In Table 57 are shown the results of this study.

The results are very dramatic. A definite bias exists between the two methods. On this group of fuels the Precision Method results

TABLE 55. ELECTRICAL CONDUCTIVITY OF FUELS

Sample Identification	Antistatic Additive	Date Prepared	Date Measured	Corrosion Inhibitor Added	Conc. (lb/1000 bbl)	Conc. (mg/l)	Actual Wgt.(g)	Average Conductivity (nS/m)	Average % Change[a]	Peak Reading IN pS/M	% Change[b]	Fuel Temp (°C)
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-02-84	None	-	-	-	424	-	-	-	22.8
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-05-84	None	-	-	-	541	-	548	-	23.3
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-07-84	None	-	-	-	534	-	543	-	23.1
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-03-84	None	-	-	-	527	-	534	-	23.2
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-12-84	None	-	-	-	529	-	534	-	22.6
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-12-84	None	-	-	-	543	-	548	-	23.1
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-14-84	None	-	-	-	546	-	548	-	24.0
83-POSF-0708 Base Fuel	Stadis 450	2-29-84	3-16-84	None	-	-	-	550	-	554	-	24.6
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-05-84	None	-	-	-	548	-	543	-	23.3
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-07-84	None	-	-	-	536	-	541	-	23.0
83-POSF-0708 Base Control	Stadis 450	2-23-84	3-08-84	None	-	-	-	529	-	541	-	23.2
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-09-84	None	-	-	-	534	-	534	-	23.1
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-12-84	None	-	-	-	532	-	534	-	22.8
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-13-84	None	-	-	-	540	-	541	-	23.1
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-14-84	None	-	-	-	536	-	541	-	23.7
83-POSF-0708 Base Control	Stadis 450	2-29-84	3-16-84	None	-	-	-	534	-	534	-	23.9
83-POSF-0703	Stadis 450	2-29-84	3-05-84	APOLLO PRI-19	9	22.8	0.0227	397	-27	397	-28	23.1
83-POSF-0703	Stadis 450	2-29-84	3-05-84	LUBRIZOL 541	6	17.1	0.0170	450	-16	450	-16	22.9
83-POSF-0708	Stadis 450	2-29-84	3-05-84	HITEC E-515	16	45.6	0.0445	198	-64	205	-63	22.7
83-POSF-0708	Stadis 450	3-06-84	3-07-84	HITEC E-515	14	39.9	0.0399	193	-64	193	-64	23.0
83-POSF-0708	Stadis 450	3-07-84	3-08-84	HITEC E-515	12	34.2	0.0341	190	-64	192	-64	22.6
83-POSF-0708	Stadis 450	3-08-84	3-09-84	HITEC E-515	10	28.5	0.0284	190	-64	192	-64	23.0
83-POSF-0708	Stadis 450	3-12-84	3-13-84	HITEC E-515	8	22.8	0.0227	200	-63	205	-63	23.3
83-POSF-0708	Stadis 450	3-13-84	3-14-84	HITEC E-515	7.5	21.4	0.0213	200	-63	205	-62	23.6
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-02-84	None	-	-	-	431	-	445	-	22.8
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-05-84	None	-	-	-	459	-	459	-	23.3
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-07-84	None	-	-	-	466	-	466	-	23.0
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-03-84	None	-	-	-	459	-	459	-	22.7
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-09-84	None	-	-	-	459	-	459	-	22.8
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-12-84	None	-	-	-	452	-	452	-	22.6
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-13-84	None	-	-	-	454	-	459	-	23.2
83-POSF-0883 Base Fuel	Stadis 450	2-29-84	3-14-84	None	-	-	-	466	-	466	-	24.0
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-16-84	None	-	-	-	463	-	466	-	24.1
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-05-84	None	-	-	-	461	-	466	-	23.3
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-07-84	None	-	-	-	466	-	466	-	23.3
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-08-84	None	-	-	-	464	-	466	-	22.8
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-09-84	None	-	-	-	466	-	466	-	22.6
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-12-84	None	-	-	-	469	-	472	-	22.8
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-13-84	None	-	-	-	461	-	466	-	22.7
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-14-84	None	-	-	-	481	-	496	-	24.2
83-POSF-0883 Base Control	Stadis 450	2-29-84	3-16-84	None	-	-	-	479	-	479	-	23.9
83-POSF-0883	Stadis 450	2-29-84	3-05-84	APOLLO PRI-19	8	22.8	0.0228	335	-27	342	-26	23.0
83-POSF-0883	Stadis 450	2-29-84	3-05-84	LUBRIZOL 541	6	17.1	0.0172	363	-21	363	-22	23.2
83-POSF-0883	Stadis 450	2-29-84	3-05-84	HITEC E-515	16	45.6	0.0455	157	-66	157	-66	23.4
83-POSF-0883	Stadis 450	3-05-84	3-07-84	HITEC E-515	14	39.9	0.0398	151	-68	151	-68	22.7
83-POSF-0883	Stadis 450	3-07-84	3-08-84	HITEC E-515	12	34.2	0.0341	153	-67	157	-66	22.7
83-POSF-0883	Stadis 450	3-08-84	3-08-84	HITEC E-515	10	28.5	0.0284	162	-65	164	-65	22.8
83-POSF-0883	Stadis 450	3-12-84	3-13-84	HITEC E-515	8	22.8	0.0227	176	-62	178	-62	23.0
83-POSF-0883	Stadis 450	3-13-84	3-14-84	HITEC E-515	7.5	21.4	0.0213	178	-62	178	-63	24.0

[a] Average % change is calculated using the average conductivity readings of the base fuel and base control on a given day as the reference point.

e.g. 83-POSF-0708, Apollo PRI-19, 3/5/84 $\left[397 \div \left(\dfrac{541 + 543}{2}\right) - 397\right] \times 100 = 27\%$ decrease

[b] % change is calculated using the average of the peak readings of the base fuel and base control on a given day as the reference point.

e.g. 83-POSF-0708, Apollo PRI-19, 3/5/84 $\left[397 \div \left(\dfrac{548 + 543}{2}\right) - 397\right] \times 100 = 28\%$ decrease

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TABLE 55 (Cont'd)

Sample Identification	Antistatic Additive	Date Prepared	Date Measured	Corrosion Inhibitor Added	Conc. (lb/1000 bbl)	Conc. (mg/l)	Actual Wgt. (g)	Average Conductivity (pS/m)	Average % Change[a]	Peak Reading IN pS/m	% Change[b]	Fuel Temp.[b] (°C)
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-02-84	None	-	-	-	548	-	548	-	23.1
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-06-84	None	-	-	-	556	-	561	-	22.7
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-07-84	None	-	-	-	573	-	575	-	22.5
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-08-84	None	-	-	-	561	-	561	-	22.5
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-09-84	None	-	-	-	548	-	548	-	21.1
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-12-84	None	-	-	-	541	-	541	-	21.6
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-13-84	None	-	-	-	556	-	561	-	22.9
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-14-84	None	-	-	-	561	-	561	-	23.1
83-POSF-1329 Base Fuel	Stadis 450	3-01-84	3-16-84	None	-	-	-	570	-	575	-	23.9
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-06-84	None	-	-	-	556	-	561	-	23.2
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-07-84	None	-	-	-	568	-	575	-	22.9
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-08-84	None	-	-	-	554	-	554	-	23.0
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-09-84	None	-	-	-	548	-	548	-	22.6
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-12-84	None	-	-	-	541	-	541	-	22.4
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-13-84	None	-	-	-	539	-	541	-	23.0
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-14-84	None	-	-	-	539	-	541	-	23.4
83-POSF-1329 Base Control	Stadis 450	3-01-84	3-16-84	None	-	-	-	541	-	548	-	23.7
	Stadis 450	3-01-84	3-06-84	APOLLO PRI-19	8	22.8	0.0228	372	-33	383	-32	23.2
	Stadis 450	3-01-84	3-06-84	LUBRIZOL 541	6	17.1	0.0171	493	-11	507	-10	22.9
	Stadis 450	3-06-84	3-07-84	HITEC E-515	16	45.6	0.0454	169	-70	178	-68	23.0
	Stadis 450	3-07-84	3-08-84	HITEC E-515	14	39.9	0.0397	180	-68	185	-68	23.0
	Stadis 450	3-08-84	3-09-84	HITEC E-515	12	34.2	0.0340	180	-68	185	-67	22.8
	Stadis 450	3-12-84	3-13-84	HITEC E-515	10	28.5	0.0283	196	-67	185	-66	22.9
	Stadis 450	3-13-84	3-14-84	HITEC E-515	8	22.8	0.0226	196	-64	205	-63	23.5
	Stadis 450			HITEC E-515	7.5	21.4	0.0212	200	-64	205	-63	23.1
83-POSF-1333 Base Fuel	Stadis 450	3-02-84	3-06-84	None	-	-	-	376	-	376	-	22.7
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-07-84	None	-	-	-	333	-	356	-	22.5
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-08-84	None	-	-	-	322	-	335	-	22.6
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-09-84	None	-	-	-	322	-	335	-	22.0
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-12-84	None	-	-	-	317	-	329	-	21.5
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-13-84	None	-	-	-	322	-	329	-	21.4
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-14-84	None	-	-	-	331	-	342	-	23.0
83-POSF-1333 Base Fuel	Stadis 450	3-01-84	3-16-84	None	-	-	-	344	-	349	-	23.0
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-06-84	None	-	-	-	342	-	342	-	23.3
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-07-84	None	-	-	-	356	-	370	-	22.5
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-08-84	None	-	-	-	342	-	356	-	22.8
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-09-84	None	-	-	-	342	-	356	-	22.9
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-12-84	None	-	-	-	335	-	342	-	22.6
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-13-84	None	-	-	-	335	-	342	-	22.4
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-14-84	None	-	-	-	340	-	356	-	22.8
83-POSF-1333 Base Control	Stadis 450	3-01-84	3-16-84	None	-	-	-	347	-	356	-	23.4
83-POSF-1333	Stadis 450	3-01-84	3-06-84	APOLLO PRI-19	8	22.8	0.0229	349	-24	356	-26	24.3
83-POSF-1333	Stadis 450	3-01-84	3-06-84	LUBRIZOL 541	6	17.1	0.0171	262	-28	267	-32	22.9
83-POSF-1333	Stadis 450	3-06-84	3-07-84	HITEC E-515	16	45.6	0.0456	246	-52	246	-55	22.7
83-POSF-1333	Stadis 450	3-07-84	3-08-84	HITEC E-515	14	39.9	0.0399	164	-51	171	-51	22.6
83-POSF-1333	Stadis 450	3-08-84	3-09-84	HITEC E-515	12	34.2	0.0342	164	-53	164	-53	22.7
83-POSF-1333	Stadis 450	3-12-84	3-13-84	HITEC E-515	10	28.5	0.0285	157	-53	157	-53	22.5
83-POSF-1333	Stadis 450	3-13-84	3-14-84	HITEC E-515	8	22.8	0.0228	153	-54	157	-54	23.1
83-POSF-1333	Stadis 450			HITEC E-515	7.5	21.4	0.0214	162	-53	164	-53	23.2

TABLE 55 (Cont'd)

Sample Identification	Antistatic Additive	Date Prepared	Date Measured	Corrosion Inhibitor Added	Conc. (lb/1000 bbl)	Conc. (mg/l)	Actual Wgt. (g)	Average Conductivity (pS/m)	Average % Change[a]	Peak Reading IN pS/M	% Change[b]	Fuel Temp.[b] (°C)
83-POSF-0708 Base Fuel	ASA-3	3-01-84	3-02-84	None	-	-	-	534	-	554	-	22.6
83-POSF-0708 Base Fuel	ASA-3	3-01-84	3-06-84	None	-	-	-	616	-	630	-	22.5
83-POSF-0708 Base Fuel	ASA-3	3-01-84	3-07-84	None	-	-	-	630	-	643	-	22.9
83-POSF-0708 Base Control	ASA-3	3-01-84	3-06-84	None	-	-	-	630	-	630	-	22.8
83-POSF-0708 Base Control	ASA-3	3-01-84	3-07-84	None	-	-	-	623	-	630	-	22.9
83-POSF-0708 Base Control	ASA-3	3-01-84	3-16-84	None	-	-	-	557	-	582	-	24.4
83-POSF-0708	ASA-3	3-01-84	3-06-84	APOLLO PRI-19	8	22.8	0.0227	616	-1	616	-2	23.2
83-POSF-0708	ASA-3	3-01-84	3-06-84	LUBRIZOL 541	6	17.1	0.0171	890	+43	890	+41	22.8
93-POSF-0708	ASA-3	3-06-84	3-07-84	LUBRIZOL 541	5	14.2	0.0142	845	+30	890	+40	22.9
83-POSF-0703	ASA-3	3-01-84	3-06-84	HITEC E-515	16	45.6	0.0455	548	-12	548	-13	23.0
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-02-84	None	-	-	-	383	-	404	-	22.5
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-06-84	None	-	-	-	386	-	404	-	22.7
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-07-84	None	-	-	-	411	-	411	-	22.5
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-08-84	None	-	-	-	390	-	397	-	22.2
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-09-84	None	-	-	-	385	-	397	-	21.5
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-12-84	None	-	-	-	356	-	376	-	21.7
83-POSF-0883 Base Fuel	ASA-3	3-01-84	3-16-84	None	-	-	-	363	-	376	-	22.3
83-POSF-0883 Base Control	ASA-3	3-01-84	3-06-84	None	-	-	-	424	-	438	-	23.1
83-POSF-0883 Base Control	ASA-3	3-01-84	3-07-84	None	-	-	-	431	-	438	-	22.9
83-POSF-0883 Base Control	ASA-3	3-01-84	3-08-84	None	-	-	-	414	-	424	-	22.9
83-POSF-0883 Base Control	ASA-3	3-01-84	3-09-84	None	-	-	-	423	-	438	-	22.7
83-POSF-0883 Base Control	ASA-3	3-01-84	3-12-84	None	-	-	-	399	-	418	-	23.3
83-POSF-0883 Base Control	ASA-3	3-01-84	3-16-84	None	-	-	-	392	-	404	-	23.2
83-POSF-0883	ASA-3	3-01-84	3-06-84	APOLLO PRI-19	8	22.8	0.0227	436	+8	438	+4	23.2
83-POSF-0883	ASA-3	3-01-84	3-06-84	LUBRIZOL 541	6	17.1	0.0171	623	+55	630	+50	23.2
83-POSF-0883	ASA-3	3-06-84	3-07-04	LUBRIZOL 541	5	14.2	0.0142	630	+50	630	+43	22.6
83-POSF-0883	ASA-3	3-07-84	3-08-84	LUBRIZOL 541	4	11.4	0.0113	628	+54	637	+55	23.0
83-POSF-0883	ASA-3	3-08-84	3-09-84	LUBRIZOL 541	3	9.55	0.00850	625	+54	637	+52	22.7
83-POSF-0883	ASA-3	3-01-84	3-06-84	HITEC E-515	16	45.6	0.0457	347	-14	356	-15	22.9

TABLE 55 (Cont'd)

Sample Identification	Antistatic Additive	Date Prepared	Date Measured	Corrosion Inhibitor Added	Conc. (lb/1000 bbl)	Conc. (mg/l)	Actual Wgt. (g)	Average Conductivity (pS/m)	Average % Change[a]	Peak Reading 1M pS/M	% Change[b]	Fuel Temp.[b] (°C)
83-POSF-1329 Base Fuel	ASA-3	2-29-84	3-02-84	None	-	-	-	513	-	534	-	23.4
83-POSF-1329 Base Fuel	ASA-3	2-29-84	3-05-84	None	-	-	-	589	-	589	-	23.3
83-POSF-1329 Base Fuel	ASA-3	2-29-84	3-07-84	None	-	-	-	573	-	575	-	23.0
83-POSF-1329 Base Fuel	ASA-3	2-29-84	3-16-84	None.	-	-	-	520	-	548	-	24.5
83-POSF-1329 Base Control	ASA-3	2-29-84	3-05-84	None	-	-	-	561	-	568	-	23.5
83-POSF-1329 Base Control	ASA-3	2-29-84	3-07-84	None	-	-	-	539	-	541	-	23.5
83-POSF-1329 Base Control	ASA-3	2-29-84	3-16-84	None	-	-	-	449	-	479	-	22.9
83-POSF-1329	ASA-3	2-29-84	3-05-84	APOLLO PRI-19	8	22.8	0.0228	466	-19	472	-18	23.6
83-POSF-1329	ASA-3	2-29-84	3-05-84	LUBRIZOL 541	6	17.1	0.0171	822	+43	856	+48	23.4
83-POSF-1329	ASA-3	2-29-84	3-07-84	LUBRIZOL 541	5	14.2	0.0142	753	+35	753	+35	23.2
83-POSF-1329	ASA-3	2-29-84	3-05-84	HITEC E-515	16	45.6	0.0456	466	-19	466	-19	23.2
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-02-84	None	-	-	-	554	-	582	-	23.6
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-05-84	None	-	-	-	958	-	958	-	23.0
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-07-84	None	-	-	-	1119	-	1232	-	22.6
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-08-84	None	-	-	-	1050	-	1095	-	22.6
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-09-84	None	-	-	-	913	-	958	-	22.4
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-12-84	None	-	-	-	667	-	685	-	22.6
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-13-84	None	-	-	-	639	-	650	-	23.2
83-POSF-1333 Base Fuel	ASA-3	2-29-84	3-14-84	None	-	-	-	748	-	821	-	23.9
83-POSF-1333 Base Control	ASA-3	2-29-84	3-05-84	None	-	-	-	765	-	822	-	23.6
83-POSF-1333 Base Control	ASA-3	2-29-84	3-07-84	None	-	-	-	876	-	958	-	22.8
83-POSF-1333 Base Control	ASA-3	2-29-84	3-08-84	None	-	-	-	1050	-	1164	-	22.8
83-POSF-1333 Base Control	ASA-3	2-29-84	3-09-84	None	-	-	-	856	-	890	-	22.8
83-POSF-1333 Base Control	ASA-3	2-29-84	3-12-84	None	-	-	-	879	-	924	-	22.6
83-POSF-1333 Base Control	ASA-3	2-29-84	3-13-84	None	-	-	-	698	-	753	-	23.2
83-POSF-1333 Base Control	ASA-3	2-29-84	3-14-84	None	-	-	-	648	-	657	-	23.9
83-POSF-1333 Base Control	ASA-3	2-29-84	3-16-84	None	-	-	-	730	-	787	-	23.4
83-POSF-1333	ASA-3	2-29-84	3-05-84	APOLLO PRI-19	8	22.8	0.0228	719	-3	753	+7	22.9
83-POSF-1333	ASA-3	2-29-84	3-05-84	LUBRIZOL 541	6	17.1	0.0170	890	+24	890	+36	23.0
83-POSF-1333	ASA-3	2-29-84	3-05-84	HITEC E-515	16	45.6	0.0457	1232	+72	1301	+64	22.9
83-POSF-1333	ASA-3	3-05-84	3-07-84	HITEC E-515	14	39.9	0.0400	1575	+52	1575	+37	22.7
83-POSF-1333	ASA-3	3-07-84	3-08-84	HITEC E-515	12	34.2	0.0343	1643	+71	1643	+55	22.2
83-POSF-1333	ASA-3	3-08-84	3-09-84	HITEC E-515	10	28.5	0.0286	1632	+82	1643	+75	22.7
83-POSF-1333	ASA-3	3-12-84	3-13-84	HITEC E-515	8	22.8	0.0228	1598	+147	1643	+151	23.1
83-POSF-1333	ASA-3	3-13-84	3-14-84	HITEC E-515	7.5	21.4	0.0214	1563	+112	1609	+100	23.9

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TABLE 55 (Cont'd)

Sample Identification	Antistatic Additive	Date Prepared	Date Measured	Corrosion Inhibitor Added	Conc. (lb/1000 bbl)	Conc. (mg/l)	Actual Wgt. (g)	Average Conductivity (pS/m)	Average % Change[a]	Peak Reading in pS/M	% Change[b]	Fuel Temp.[b] (°C)
82-POSF-0113 Base Fuel	ASA-3	3-01-84	3-02-84	None	-	-	-	452	-	466	-	23.0
82-POSF-0113 Base Fuel	ASA-3	3-01-84	3-06-84	None	-	-	-	495	-	500	-	22.7
82-POSF-0113 Base Fuel	ASA-3	3-01-84	3-07-84	None	-	-	-	479	-	479	-	22.4
82-POSF-0113 Base Fuel	ASA-3	3-01-84	3-13-84	None	-	-	-	457	-	466	-	22.5
82-POSF-0113 Base Fuel	ASA-3	3-01-84	3-14-84	None	-	-	-	459	-	466	-	23.1
82-POSF-0113 Base Fuel	ASA-3	3-01-84	3-16-84	None	-	-	-	445	-	466	-	23.4
82-POSF-0113 Base Control	ASA-3	3-01-84	3-06-84	None	-	-	-	486	-	500	-	22.5
82-POSF-0113 Base Control	ASA-3	3-01-84	3-07-84	None	-	-	-	477	-	479	-	22.8
82-POSF-0113 Base Control	ASA-3	3-01-84	3-13-84	None	-	-	-	454	-	456	-	23.3
82-POSF-0113 Base Control	ASA-3	3-01-84	3-14-84	None	-	-	-	461	-	472	-	23.8
8C-POSF-0113 Base Control	ASA-3	3-01-84	3-16-84	None	-	-	-	449	-	472	-	24.5
82-POSF-0113	ASA-3	3-01-84	3-06-84	APOLLO PRI-19	8	22.8	0.0229	404	-18	404	-19	22.5
82-POSF-0113	ASA-3	3-01-84	3-06-84	LUBRIZOL 541	6	17.1	0.0170	708	+44	719	+44	22.0
82-POSF-0113	ASA-3	3-06-84	3-07-84	LUBRIZOL 541	4	14.2	0.0142	719	+50	719	+50	22.8
82-POSF-0113	ASA-3	3-07-84	3-13-84	LUBRIZOL 541	4	11.4	0.0113	669	+47	685	+47	23.0
82-POSF-0113	ASA-3	3-13-84	3-14-84	LUBRIZOL 541	3	8.55	0.00848	657	+43	671	+43	23.5
82-POSF-0113	ASA-3	3-01-84	3-06-84	HITEC E-515	16	45.6	0.0456	466	-5	466	-4	22.4
83-POSF-1330 Base Fuel	ASA-3	3-01-84	3-02-84	None	-	-	-	424	-	438	-	22.7
83-POSF-1330 Base Fuel	ASA-3	3-01-84	3-06-84	None	-	-	-	481	-	486	-	22.8
83-POSF-1330 Base Fuel	ASA-3	3-01-84	3-07-84	None	-	-	-	493	-	493	-	22.6
83-POSF-1330 Base Fuel	ASA-3	3-01-84	3-14-84	None	-	-	-	437	-	449	-	23.2
83-POSF-1330 Base Fuel	ASA-3	3-01-84	3-16-84	None	-	-	-	447	-	472	-	24.1
83-POSF-1330 Base Control	ASA-3	3-01-84	3-07-84	None	-	-	-	468	-	472	-	22.8
83-POSF-1330 Base Control	ASA-3	3-01-84	3-14-84	None	-	-	-	469	-	472	-	23.0
83-POSF-1330 Base Control	ASA-3	3-01-84	3-16-84	None	-	-	-	413	-	424	-	23.5
83-POSF-1330 Base Control	ASA-3	3-01-84	3-06-84	None	-	-	-	395	-	411	-	23.3
83-POSF-1330	ASA-3	3-01-84	3-06-84	APOLLO PRI-19	8	22.8	0.0229	445	-6	452	-6	23.0
83-PCSF-1330	ASA-3	3-01-84	3-06-84	LUBRIZOL 541	6	17.1	0.0171	671	+42	678	+42	23.0
83-POSF-1330	ASA-3	3-06-84	3-07-84	LUBRIZOL 541	5	14.2	0.0142	664	+38	671	+39	23.9
83-POSF-1330	ASA-3	3-13-84	3-14-84	LUBRIZOL 541	4	11.4	0.0114	563	+32	568	+30	23.9
83-POSF-1330	ASA-3	3-01-84	3-06-84	HITEC E-515	16	45.6	0.0457	397	-16	397	-15	22.9

TABLE 55 (Cont'd)

Sample Identification	Antistatic Additive	Date Prepared	Date Measured	Corrosion Inhibitor Added	Conc. (lb/1000 bbl)	Conc. (mg/l)	Actual Wgt. (g)	Average Conductivity (pS/m)	Average % Change[a]	Peak Reading IN pS/M	% Change[b]	Fuel Temp.[b] (°C)
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-02-84	None	-	-	-	520	-	520	-	-
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-05-84	None	-	-	-	520	-	520	-	23.3
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-07-84	None	-	-	-	507	-	534	-	22.8
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-08-84	None	-	-	-	516	-	527	-	23.1
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-09-84	None	-	-	-	527	-	527	-	23.1
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-12-84	None	-	-	-	534	-	534	-	23.0
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-13-84	None	-	-	-	535	-	541	-	23.1
82-POSF-0113 Base Fuel	Stadis 450	2-29-84	3-14-84	None	-	-	-	534	-	541	-	23.7
82-POSF-0113 Base Control	Stadis 450	2-29-84	3-16-84	None	-	-	-	534	-	534	-	23.2
82-POSF-0113 Base Control	Stadis 450	2-29-84	3-05-84	None	-	-	-	513	-	520	-	22.8
82-POSF-0113 Base Control	Stadis 450	2-29-84	3-07-84	None	-	-	-	500	-	520	-	22.8
82-POSF-0113 Base Control	Stadis 450	2-29-84	3-08-84	None	-	-	-	461	-	493	-	22.8
82-POSF-0113 Base Control	Stadis 450	2-29-84	3-12-84	None	-	-	-	483	-	493	-	23.2
82-POSF-0113 Base Control	Stadis 450	2-29-84	3-13-84	None	-	-	-	477	-	479	-	23.1
82-POSF-0113	Stadis 450	2-29-84	3-16-84	None	-	-	-	470	-	472	-	23.1
82-POSF-0113	Stadis 450	2-29-84	3-05-84	APOLLO PRI-19	8	22.8	0.0227	342	-34	343	-33	23.0
82-POSF-0113	Stadis 450	2-29-84	3-05-84	LUBRIZOL 541	6	17.1	0.0172	424	-18	431	-17	22.8
82-POSF-0113	Stadis 450	2-29-84	3-05-84	HITEC E-515	16	45.6	0.0456	151	-71	157	-71	22.9
82-POSF-0113	Stadis 450	3-05-84	3-07-84	HITEC E-515	14	39.9	0.0399	153	-70	157	-70	23.0
82-POSF-0113	Stadis 450	3-07-84	3-08-84	HITEC E-515	12	34.2	0.0342	146	-70	151	-70	23.2
82-POSF-0113	Stadis 450	3-03-84	3-09-84	HITEC E-515	10	28.5	0.02850	146	-70	151	-70	23.0
82-POSF-0113	Stadis 450	3-12-84	3-13-84	HITEC E-515	8	22.8	0.0228	164	-68	164	-66	23.2
82-POSF-0113	Stadis 450	3-13-84	3-14-84	HITEC E-515	7.5	21.4	0.0214	164	-68	164	-63	23.1
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-02-84	None	-	-	-	561	-	568	-	23.2
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-07-84	None	-	-	-	575	-	575	-	23.5
83-POSF-1330 Base Fuel	Stadis 450	3-05-84	3-08-84	None	-	-	-	573	-	589	-	23.5
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-09-84	None	-	-	-	580	-	592	-	23.2
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-12-84	None	-	-	-	534	-	534	-	23.0
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-13-84	None	-	-	-	573	-	575	-	22.8
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-14-84	None	-	-	-	536	-	541	-	23.6
83-POSF-1330 Base Fuel	Stadis 450	2-29-84	3-16-84	None	-	-	-	587	-	589	-	23.7
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-05-84	None	-	-	-	587	-	589	-	24.6
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-07-84	None	-	-	-	554	-	554	-	22.8
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-08-84	None	-	-	-	554	-	561	-	23.4
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-09-84	None	-	-	-	543	-	548	-	23.1
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-12-84	None	-	-	-	525	-	541	-	22.8
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-13-84	None	-	-	-	543	-	548	-	22.9
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-13-84	None	-	-	-	536	-	541	-	23.5
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-16-84	None	-	-	-	543	-	546	-	23.6
83-POSF-1330 Base Control	Stadis 450	2-29-84	3-16-84	None	-	-	-	550	-	554	-	24.2
83-POSF-1330	Stadis 450	3-05-84	3-05-84	APOLLO PRI-19	8	22.8	0.0230	397	-30	397	-29	23.1
83-POSF-1330	Stadis 450	2-29-84	3-05-84	LUBRIZOL 541	6	17.1	0.0171	502	-11	507	-10	23.1
83-POSF-1330	Stadis 450	3-05-84	3-05-84	HITEC E-515	16	45.6	0.0454	185	-67	185	-67	23.4
83-POSF-1330	Stadis 450	3-07-84	3-07-94	HITEC E-515	14	39.9	0.0397	178	-68	178	-69	23.1
83-POSF-1330	Stadis 450	3-07-84	3-08-84	HITEC E-515	12	34.2	0.0340	169	-70	171	-70	22.9
83-POSF-1330	Stadis 450	3-12-84	3-13-84	HITEC E-515	10	28.5	0.0283	176	-67	178	-67	22.8
83-POSF-1330	Stadis 450	3-13-84	3-13-84	HITEC E-515	8	22.8	0.0226	185	-65	185	-66	23.7
83-POSF-1330	Stad 950	3-13-84	3-14-84	HITEC E-515	7.5	21.4	0.0212	190	-66	192	-66	23.6

Table 56. SUMMARY OF FINDINGS

Sample I.D.	Stadis 450	ASA-3
83-POSF-0708	Apollo and Lubrizol blends at M.A.C. <40% decrease. Hitec blend at M.E.C. >60% decrease.	Apollo and Hitec blends at M.A.C. slight decrease. Lubrizol blend near M.A.C. slightly less than 40% increase.
83-POSF-0883	Apollo and Lubrizol blends at M.A.C. <40% decrease. Hitec blend at M.E.C. >60% decrease.	Apollo and Hitec blends at M.A.C. slight change. Lubrizol blend at M.E.C. >50% increase.
83-POSF-1329	Apollo and Lubrizol blends at M.A.C. <40% decrease. Hitec blend at M.E.C. >60% decrease.	Apollo and Hitec blends at M.A.C. <40% decrease. Lubrizol blend near M.A.C. slightly less than 40% increase.
83-POSF-1333	Apollo and Lubrizol blends at M.A.C. <40% decrease. Hitec blend at M.E.C. >50% decrease.	Apollo blend at M.A.C. slight change. Lubrizol blend at M.A.C. <40% increase. Hitec blend at M.E.C. much higher than 40% increase.
82-POSF-0113	Apollo and Lubrizol blends at M.A.C. <40% decrease. Hitec blend at M.E.C. >60% decrease.	Apollo and Hitec blends at M.A.C. <40% decrease. Lubrizol blend at M.E.C. >40% increase.
83-POSF-1330	Apollo and Lubrizol blends at M.A.C. <40% decrease. Hitec blend at M.E.C. >60% decrease.	Apollo and Hitec blends at M.A.C. <40% decrease. Lubrizol blend near M.E.C. >40% increase.

Note: Increases and decreases noted, refer to electrical conductivity changes as measured by ASTM D 3114.

M.A.C. - Maximum Allowable Concentration
M.E.C. - Minimum Effective Concentration

Table 57. ELECTRICAL CONDUCTIVITY METHOD COMPARISON

Sample No.	Antistatic Additive	Date Measured	Handheld[a] Meter (pS/m)	Precision[b] Method (pS/m)	% Variation	Fuel Temp. (°C)
83-POSF-0708 Base Fuel	Stadis 450	3-26-84	380	539	+42	23.6
83-POSF-0883 Base Fuel	Stadis 450	3-23-84	343	466	+36	23.7
82-POSF-0113 Base Fuel	Stadis 450	3-23-84	400	534	+34	23.5
83-POSF-1330 Base Fuel	Stadis 450	3-23-84	423	584	+38	23.4
83-POSF-1329 Base Fuel	Stadis 450	3-23-84	420	570	+36	23.7
83-POSF-1333 Base Fuel	Stadis 450	3-26-84	210	292	+39	24.1
83-POSF-0708 Base Fuel	ASA-3	3-26-84	420	564	+34	24.0
83-POSF-0883 Base Fuel	ASA-3	3-26-84	290	397	+37	23.5
82-POSF-0113 Base Fuel	ASA-3	3-23-84	343	456	+33	23.2
83-POSF-1330 Base Fuel	ASA-3	3-26-84	323	438	+36	23.5
83-POSF-1329 Base Fuel	ASA-3	3-26-84	363	493	+36	23.3
83-POSF-1333 Base Fuel	ASA-3	3-23-84	423	579	+37	23.4

[a] ASTM D 2624 using EMCEE Electronics Meter, Model No. 1151, I.D.# JH7183
[b] ASTM D 3114

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are substantially higher than the Portable Meter Method. The differences show a range of 33 to 42% with the average at about 36.5%. A plot of the values in Table 57 is shown in Figure 18, as well as values compiled earlier by AFWAL/POSF. Also, a shale JP-4 fuel which was used as a reference for checking inter-laboratory results for the portable meter method is included in the figure.

Conclusions

1. Some definite interactions were observed between corrosion inhibitors and antistatic additives

    a. All fuels containing Stadis 450 had electrical conductivity increases >40% when Hitec E-515 was added at its minimum effective concentration.

    b. Fuels containing ASA-3 had some interaction with Lubrizol 541. Several blends were marginal; one exceeded the 40% limit at its minimum effective concentration.

    c. All fuels with both Stadis 450 and ASA-3 additives showed little effect on electrical conductivity when Apollo PRI-19 was added.

2. A bias exists between the laboratory precision method used by Monsanto, and the portable, hand-held meter method. On the fuels evaluated in this program, the precision method averaged some 36% higher than the portable meter.
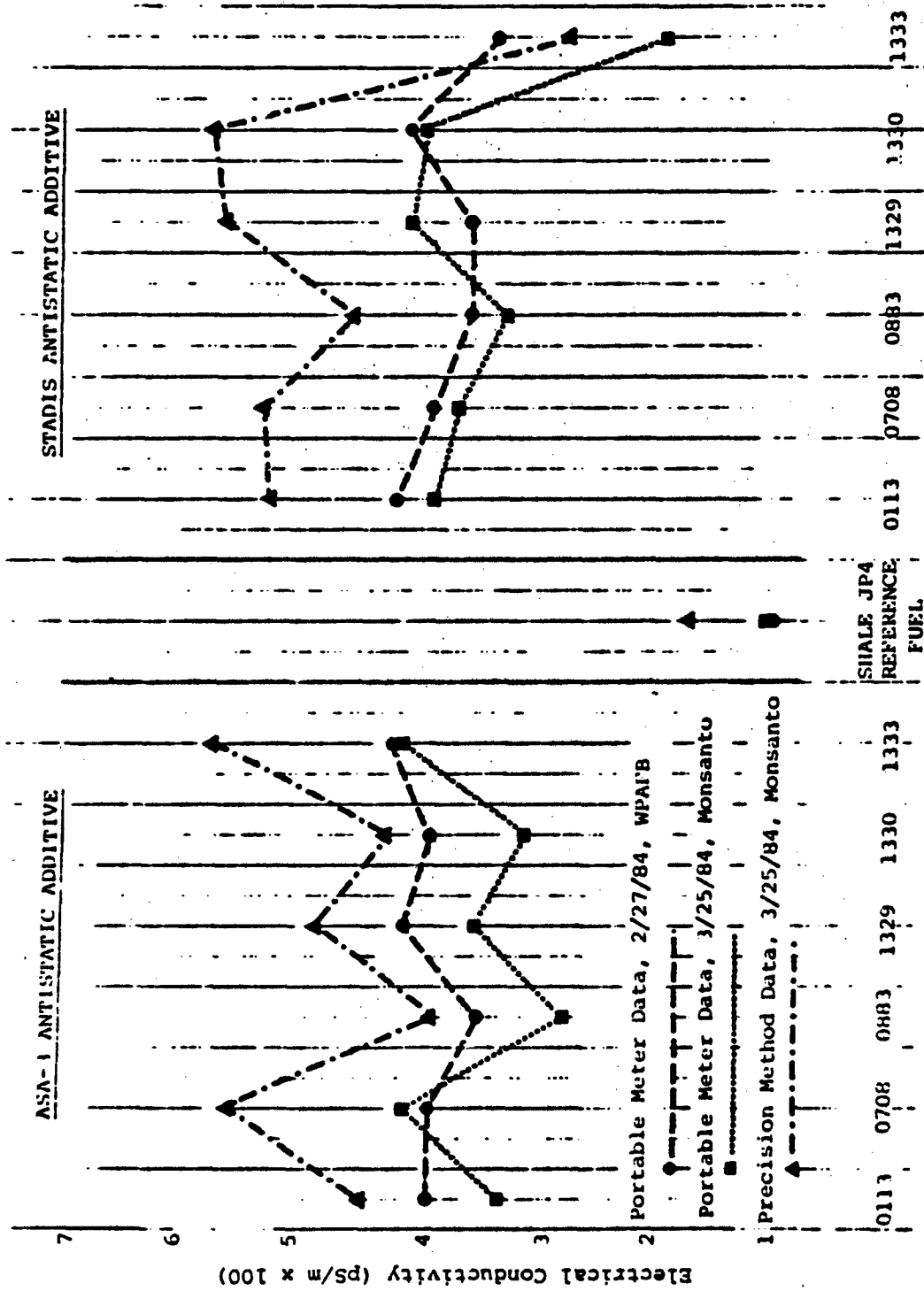
Figure 18. Comparison of Precision and Portable Meter Data with Selected Fuels

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3. INVESTIGATION OF COPPER STRIP CORROSION TEST FAILURE BY SHALE-DERIVED JP-4

A series of samples was examined to aid in determining the cause of failure of copper strip corrosion tests by shale-derived JP-4. Six fuel and naphtha samples, and copper strips used in their testing, were examined. The following samples were provided for this investigation.

83-POSF-0836	1 GALLON	2B STRIP (CORROSION ON CAP)
83-POSF-0837	1 GALLON	4B STRIP
83-POSF-1071	1 GALLON	2C STRIP
83-POSF-1168	200 ml	1B STRIP (ONLY PASSING STRIP)
83-POSF-1167	200 ml	4A STRIP
83-POSF-1184	1 QUART	2A STRIP (PRODUCTION SAMPLE)

BLANK STRIP FRESHLY POLISHED

BLANK TOWEL ONLY

CYLINDER # 174 (BOC TESTER) USED TO TEST CARIBOU SHALE JP-4

BACKGROUND INFORMATION

The first three samples listed above were naphtha samples from the Caribou refinery. Sample-1184 was a sample of shale JP-4 which failed the copper strip corrosion test and showed an unusual dark deposit along the ball-on-cylinder (BOC) wear track after lubricity testing. Samples-1167 and -1168 originated from HRI shale production. Sample-1167 failed copper strip corrosion originally and represented the first run material from HRI productions. The first run material was ultimately doped with benzotriazole to allow it to pass the corrosion test. Sample -1167, however, apparently had not been doped.

The Caribou fuel was known to have contacted, at some time or other during its processing, ammonia, hydrogen sulfide, sodium hydroxide, ethylmercaptan, carbon dioxide and water.

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Experimental

A 50-ml aliquot of each sample was extracted with two separate 25-ml portions of deionized water. The water layer was found to have a pH of slightly less than 7, suggesting that ammonia and/or sodium hydroxide were not present in the samples as contaminants. Simple computations show that sodium hydroxide present in the fuel at a level as low as 0.05 ppm by weight would result in a pH of approximately 8 (10^{-6}N NaOH) in the water extract. Higher concentrations would, of course, increase the pH proportionately.

Analysis of Copper Strips

The copper strips used in corrosivity testing of the samples were analyzed by energy dispersive x-ray fluorescence (XRF) using an EDAX 707A x-ray analyzer. Included in the analysis was the corroded metallic liner from a cap used on the container for sample 83-POSF-0836. Sulfur was specifically sought in these specimens. Results of the analyses are presented in Table 58.

TABLE 58. X-RAY ANALYSIS OF COPPER STRIPS AND CAP LINER

	Area counts					
Element/line	83-POSF-1167	83-POSF-0837	83-POSF-1168	Blank copper strip	Cap liner w/corrosion	Cap liner back side
Copper K_α	412,569	418,736	426,949	416,295	-	-
Copper K_β	58,905	60,405	61,328	60,261	-	-
Sulfur K_α	2,936	2,216	-	298	3,267	736
Tin L_{α_1}	-	-	-	-	37,620	33,758
Tin L_{β_1}	-	-	-	-	4,445	3,905
Iron K_α	-	-	-	-	47,557	582
Iron K_β	-	-	-	-	-	4,124
Chlorine K_α	-	-	-	-	-	4,124

The corroded copper strips were found to contain significant levels of sulfur. The level was approximately 10-fold greater than the small amount found on the blank strip. The copper strip used to test the fuel which passed the corrosivity test (83-POSF-1168) showed no sulfur on its surface.

The cap liner, which was made of tin, showed the presence of both iron and sulfur on its badly corroded and discolored outer surface. A small amount of sulfur and a trace of chlorine was found on the side which was attached to the lid. These latter elements may have been a part of the adhesive used in fabrication of the cap.

<u>Determination of Sulfur Compounds</u>

Based on the preceding experiments, hydrogen sulfide, mercaptans or free sulfur in the fuels were believed to be possible causes of copper corrosion. The first two of these forms of sulfur can be readily measured by a potentiometric titration described in UOP Test Method 163-80. A silver-silver sulfide electrode is used to indicate the end-point in the titration of the mentioned sulfur compounds with a standard solution of alcoholic silver nitrate. Free sulfur can be detected by the procedure but cannot be readily quantitated because of the ease with which it reacts with mercaptide ions. The titration curve, however will have three separate breaks corresponding to hydrogen sulfide, mercaptan, and free sulfur, should they be present. Detection limits for sulfide and mercaptide ions are 1.0 ppm; a detection limit for sulfur has not been established, but would be expected to be in the same range.

The procedure was used to analyze the six hydrocarbon (fuel and naphtha) samples. Test analyses were conducted to validate the procedure using a known solution (17.2 ppm sulfur) of 2-methyl, 2-propyl mercaptan in iso-octane. The slightly lower analytical value obtained (16.8 ppm) probably reflected the fact that the reference mercaptan was "practical" grade not reagent grade. Typical
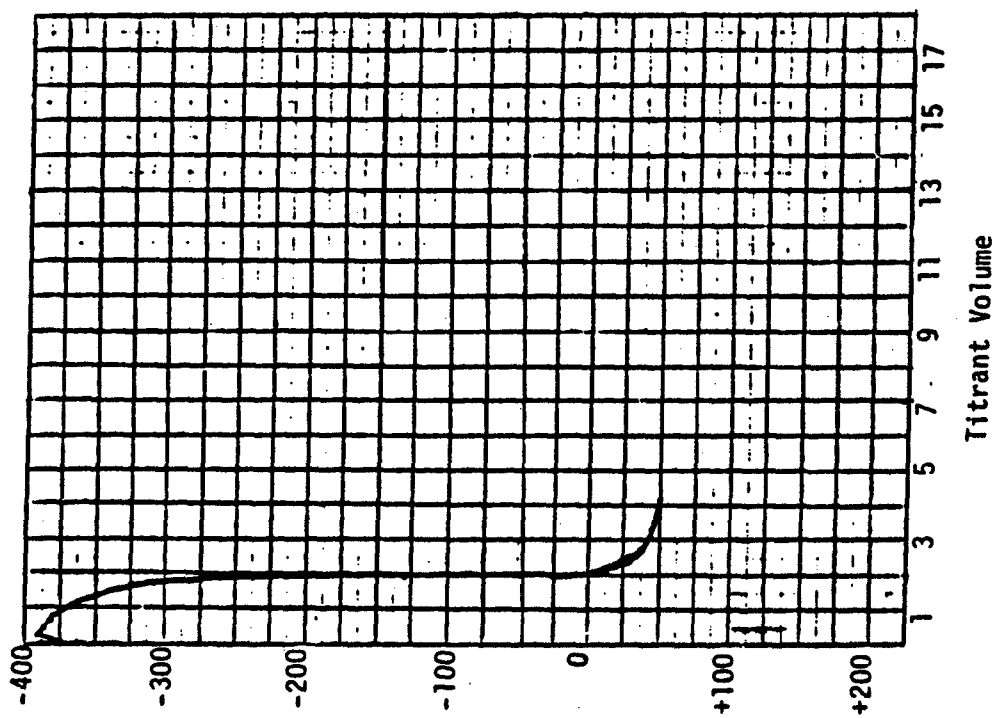
A. REFERENCE SOLUTION OF 17.2 ppm MERCAPTIDE
SULFUR IN ISO-OCTANE
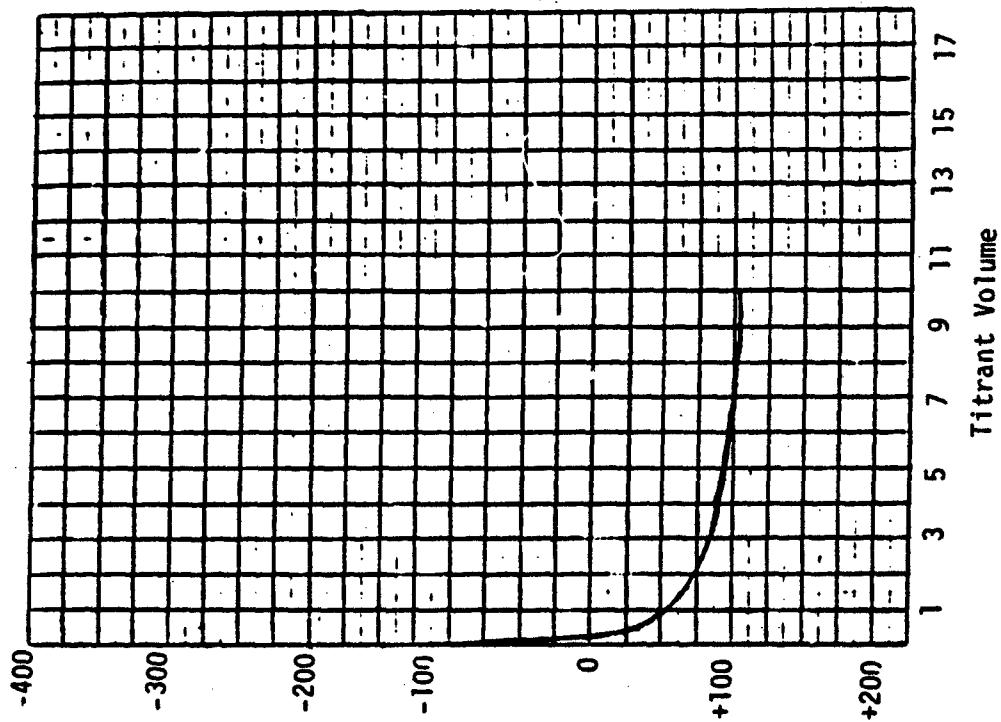
B. FUEL SAMPLE 83-POSF-1167

FIGURE 1. TYPICAL POTENTIOMETRIC TITRATION CURVES FOR MERCAPTIDE/SULFIDE IN FUELS

titration curves are shown in Figure 9. Surprisingly, no sulfur in any of the three forms was detected in the samples. It can thus be concluded that hydrogen sulfide and mercaptan sulfur were not present in the sample. No free sulfur was detected, but the detection limit is not known. Sulfur may be present in other forms such as sulfonates, sulfonic acid, thiophenes, etc.

Analysis of Ball-On-Cylinder Wear Track Debris

The BOC component, cylinder No. 174, was analyzed by energy dispersive x-ray analysis in conjunction with scanning electron microscopy (SEM) to provide information on the unusual wear-scar deposit. The sample was too large for the SEM chamber so the debris was transferred onto double stick tape. The sample was coated with a thin layer of gold/palladium to make the sample conductive. The x-ray spectrum showed that the primary element detected was iron.

In order to determine the light element content of the deposit (atomic numbers <11) the sample was analyzed using Auger Electron Spectroscopy (AES). To prevent contamination of the material during transfer, the entire cylinder was placed into the AES chamber. The sample was profiled by ion milling in order to determine composition as a function of depth. As the substrate was approached, both carbon and oxygen decreased indicating that the deposit was made up largely of carbon and oxygen with some iron also being present. No other elements were present in the deposit of debris at the cylinder wear scar.

Conclusion

The presence of significant amounts of sulfur on the corroded copper test strips strongly suggest the presence of corrosive sulfur containing materials in certain fuels. Hydrogen sulfide and

mercaptan compounds were shown not to be present. No indication
was obtained of the presence of free sulfur. Other sulfur com-
pounds (e.g. sulfonic acids, thiophenes etc.) apparently were
involved in the copper corrosion problem. The debris at the BOC
wear-track might not have been related to the copper strip cor-
sion.

4. DETERMINATION OF CAUSE OF SHALE JP-4 FUEL PROBLEMS

The Caribou Refinery Program had produced, over a 5-month period, several hundred thousand gallons of JP-4 jet fuel for engine tests, additive evaluation, and toxicology testing. Even though the fuels had passed specification tests according to MIL-T-5624L, problems which appeared to be fuel related had arisen in fuel pumps, ball-on-cylinder tests, and jet fuel thermal oxidation tests (JFTOT).

Four shale JP-4 samples from this program were initially provided for evaluation:

83-POSF-1312	Batch #1, Blend #4, Tank Samples JP-4
83-POSF-1271	Batch #1, Blend #3, Refinery Sample JP-4
83-POSF-0649	Shale U.O.P. Phase III JP-4
83-POSF-1340	Batch #2, Blend #1, Refinery JP-4

Trace Metals Analyses

These four samples of shale derived JP-4 were analyzed for trace metals using an ISA Model JY48P inductively coupled plasma (ICP) spectrometer. The ICP analyses were conducted on aqueous acid extracts (ultrapure HCl) of the fuels which provided a concentration factor of four for the metals. The test results for the fuel samples, an acid extraction blank, and a reference standard are given in Table 59. In examining the data in Table 59, it can be seen that Sample 83-POSF-1312 shows detectable levels of several metals that were not found in the other three fuels.

Elemental Analysis

Further elemental analysis for sodium and chlorine was carried out on these fuels, as well as on a number of other fuels. Results of these analyses are presented in Table 60.

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TABLE 59. TRACE METALS ANALYSES BY ICP SPECTROMETRY

Elements of detection:	Fe	Mg	Zn	Ca	Cu	B	Ba	Cr
Instrument quantification limit, LOQ, ppb:	15	3	6	140	57	140	12	27
LOQ after concentration[a], ppb:	3.8	0.8	1.5	35	14.3	35	3	6.8
Acid extraction blank[b]:	x	x	x	x	x	x	x	x
Concentration of element[c] in fuel samples, ppb								
83-POSF-0649	70	25	123	38	x	x	x	x
83-POSF-1271	148	30	170	53	x	x	x	x
83-POSF-1312	153	33	28	03	30	98	28	7
83-POSF-1340	53	19	21	x	x	x	x	x
Analysis of reference standard, ppb								
True values	1,000	1,000	1,000	10,000	1,000	1,000	10,000	1,000
Observed value	1,000	1,000	930	10,000	910	1,000	10,000	990
Percent recovery	100	100	98	100	91	100	100	99

(continued)

TABLE 59 (continued)

Elements of detection:	Pb	Si	Cd	Co	Mn	Ni	V	Al
Instrument quantification limit. LOQ, ppb:	150	400	30	45	6	27	30	55
LOQ after concentration[a], ppb:	37.5	100	7.5	11.3	1.5	6.8	7.5	13.8
Acid extraction blank[b]:	x	x	x	x	x	x	x	x
Concentration of element in fuel samples[c], ppb								
83-POSF-0649	x	x	x	x	x	x	x	x
83-POSF-1271	x	x	x	x	x	x	x	x
83-POSF-1312	85	155	x	x	x	x	x	x
83-POSF-1340	x	x	x	x	x	x	x	x
Analysis of reference standard, ppb								
True values	1,000	-	1,000	1,000	1,000	1,000	1,000	10,000
Observed value	1,000	-	1,000	990	1,000	990	1,000	9,800
Percent recovery	100	-	100	99	100	99	100	98

(continued)

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TABLE 59 (continued)

Elements of detection:	Be	Mo	Ti	P	Ag	Sn	Na	Sr	Sb
Instrument quantification limit, LOQ, ppb	2	190	36	500	190	1,500	30,000	9	300
LOQ after concentration[a], ppb:	0.5	47.5	9	125	47.5	375	7,500	2.3	75
Acid extraction blank[b]:	X	X	X	X	X	X	X	X	X
Concentration of element in fuel samples[c], ppb									
83-POSF-0649	X	X	X	X	X	X	X	X	X
83-POSF-1271	X	X	X	X	X	X	X	X	X
83-POSF-1312	X	X	X	X	X	X	X	X	X
83-POSF-1340	X	X	X	X	X	X	X	X	X
Analysis of reference standard, ppb									
True values	1,000	1,000	1,000	-	-	-	-	-	-
Observed value	990	980	990	-	-	-	-	-	-
Percent recovery	99	98	99	-	-	-	-	-	-

[a]Fuel samples were effectively concentrated by a factor of 4 for the analysis by extraction with a small volume of aqueous acid.

[b]Aqueous ultrapure acid was used to extract the fuel samples for analysis. An "X" shows that the element was not detected at the instrument LOQ.

[c]An "X" shows that the element was not detected at the LOQ after concentration. The numerical values were obtained by dividing the observed value by 4. These values represent the element concentration in the original fuels.

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TABLE 60. ELEMENTAL ANALYSES

Sample No.	Identification	Sodium,[a] ppb	Chlorine,[b] ppb
83-POSF-0649	U.O.P. shale fuel, Phase III	230	50
83-POSF-1271	Refinery shale JP-4, batch 1, blend 3	420	100
83-POSF-1312	Refinery shale JP-4, batch 1, blend 4	1300	235
83-POSF-1340	Refinery shale JP-4, batch 2, blend 1	120	200
83-POSF-1355	Shale JP-4 from P&W	110	-
83-POSF-1371	Shale JP-4 from P&W	220	-
83-POSF-1374	Shale JP-4 from P&W	130	-
83-POSF-1389	Shale JP-4, Caribou refinery, additive free	330	-
83-POSF-1390	Shale JP-4, Caribou refinery, additive free, Tk. 528	110	-
83-POSF-0928	Petroleum JP-4	-	200

[a] Determination by atomic absorption spectrometry.
[b] Analyses by specific ion electrode.

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The atomic absorption analyses for sodium were carried out on aqueous acid extracts of the fuels. High purity HCl was used, followed by water dilution so that a concentration factor of four was achieved. (Results in Table 60 are the actual sodium levels in the fuel). Chloride analyses were conducted using an Orion specific ion eletrode, and the "known addition" technique.

In examining the data, one can see nothing dramatic with regard to the chloride data. However, the sodium data are interesting, as Sample 83-POSF-1312 again is unique with a sodium value that is 3-12 times higher than any of the other fuels tested.

Chromatographic Separation and Analysis

These four samples were also separated into polar and nonpolar components by column chromatography using a silica gel column. The polar components were separated using three different eluants: (1) diethyl ether to remove slightly polar components; (2) chloroform for moderately polar components; and (3) ethanol for highly polar components. Analyses of the various polar fractions were carried out using a Hewlett Packard 5880 Gas Chromatograph equipped with a nitrogenphosphorus detector. No significant level of nitrogen compounds was found in any of the samples (detection limit for nitrogen was <1 ppm). Any trace amounts of nitrogen compounds that did appear were low-boiling, and were seen near the solvent peak.

Particulate Considerations

An attempt was made to compare the relative amounts of particulates in fuel 83-POSF-1312 with two fuels from Task No. 2035-090, 83-POSF-1261 and 83-POSF-1271, and two other fuels, 83-POSF-1281 and 83-POSF-1282. Particulate examination was done by filtering 75 mL of each fuel through a 0.45 μ Millipore filter. Each fuel was placed in a filter funnel and allowed to drip slowly on a small area of the filter; this area was then discolored by the

fuel. The most intense discoloration was observed with fuel 83-POSF-1312; the rest of the fuels in descending order of discoloration were 83-POSF-1261, -1271, -1281, and -1282.

The filters from fuels 83-POSF-1312 and 83-POSF-1261 were then submitted for elemental x-ray analysis (EDAX). The only real difference observed by this technique between the two fuels was in the amount of iron, where fuel 83-POSF-1312 showed a substantially higher level.

Analysis of Ball-On-Cylinder Wear-Track Debris

Ball and cylinder samples (83-POSF-1326 and 83-POSF-1320, respectively) were submitted for analysis of wear deposits. Initially, the surface of the ball deposit was examined by scanning electron microscopy (SEM)[a], Figure 20, and by energy dispersive x-ray analysis (EDXA). The EDXA examination showed only the Fe and Cr from the substrate. In order to analyze for the lighter elements (atomic numbers <11), Auger Electron Spectroscopy (AES)[a] was utilized. Figures 21-24 show various AES spectra of the ball and its deposits. All show the deposits to contain carbon and oxygen. In Figure 25, the ball sample was profiled and the deposit was found to be greater than 100 Å thick.

The cylinder was too large to place directly in the AES, so an alternate method of sampling the deposit was utilized. Indium foil was rubbed across the deposit, and the foil containing transferred deposit was mounted in the AES sample tray. Figure 26 has the spectrum from this analysis. Again, carbon and oxygen are present; and, of course, indium. The small amount of iron in the transferred deposit may be indicative of some iron tied up in the deposit, or that some of the substrate materials was transferred.

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[a]Analyses were carried out at the University of Dayton Research Institute, Metals and Ceramics Division.
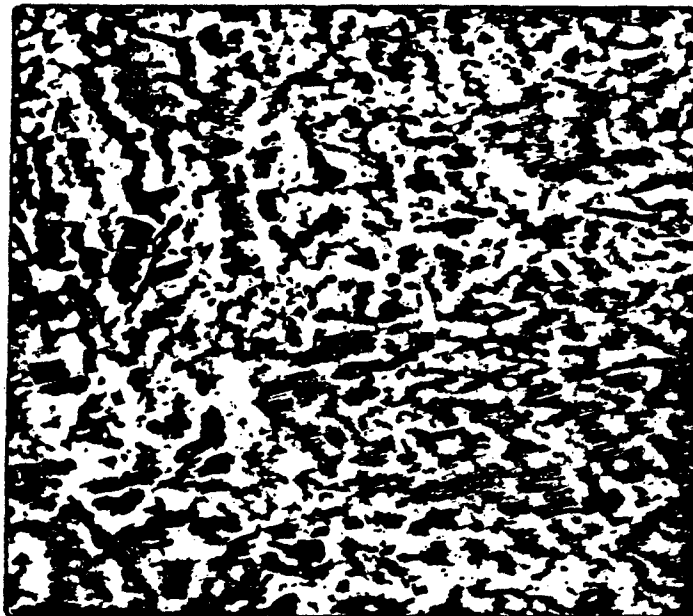
Figure 20.   SEM of ball deposit (500X).
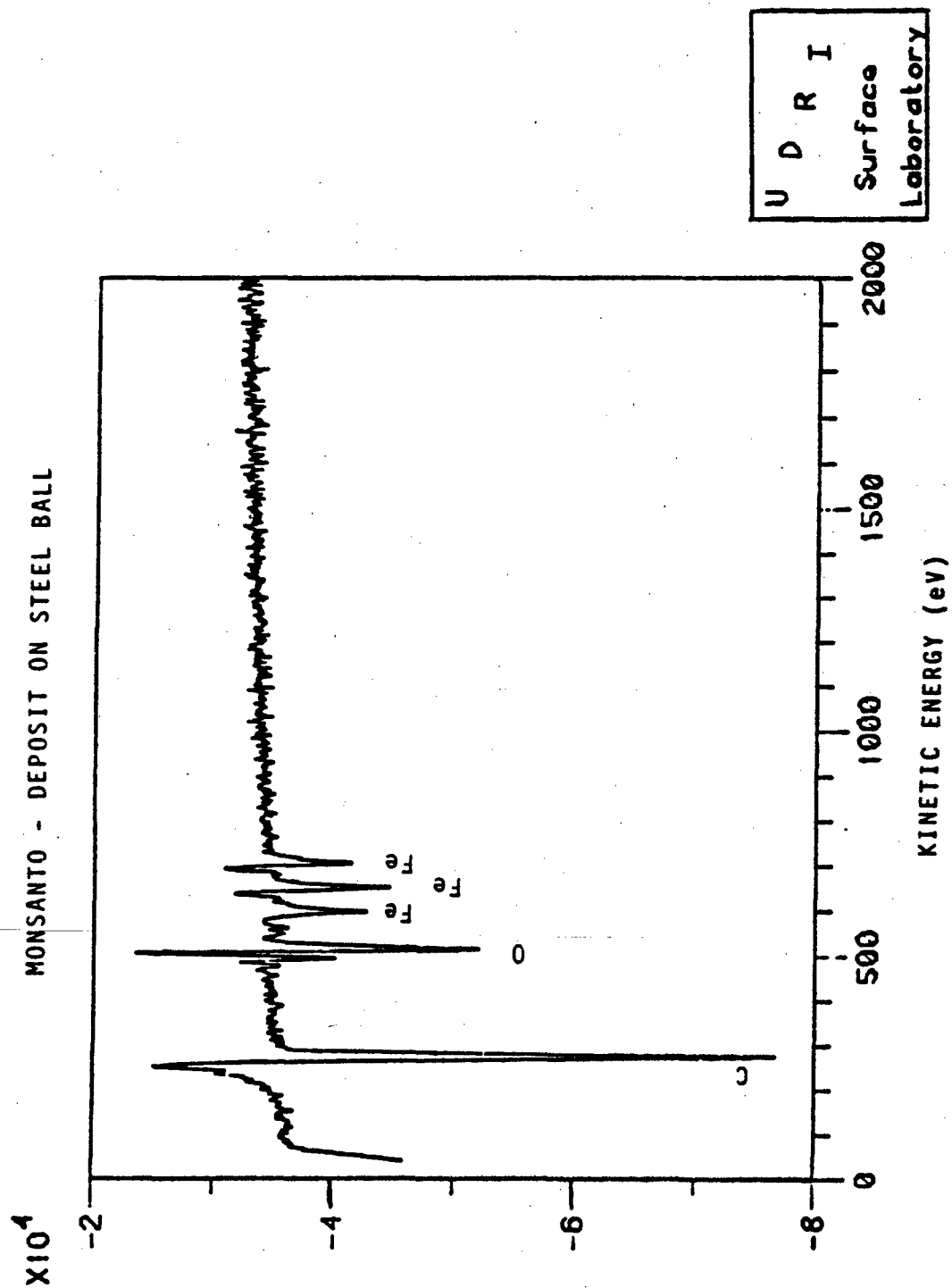
Figure 21. Analysis by AES of deposit on steel ball (83-POSF-1326).
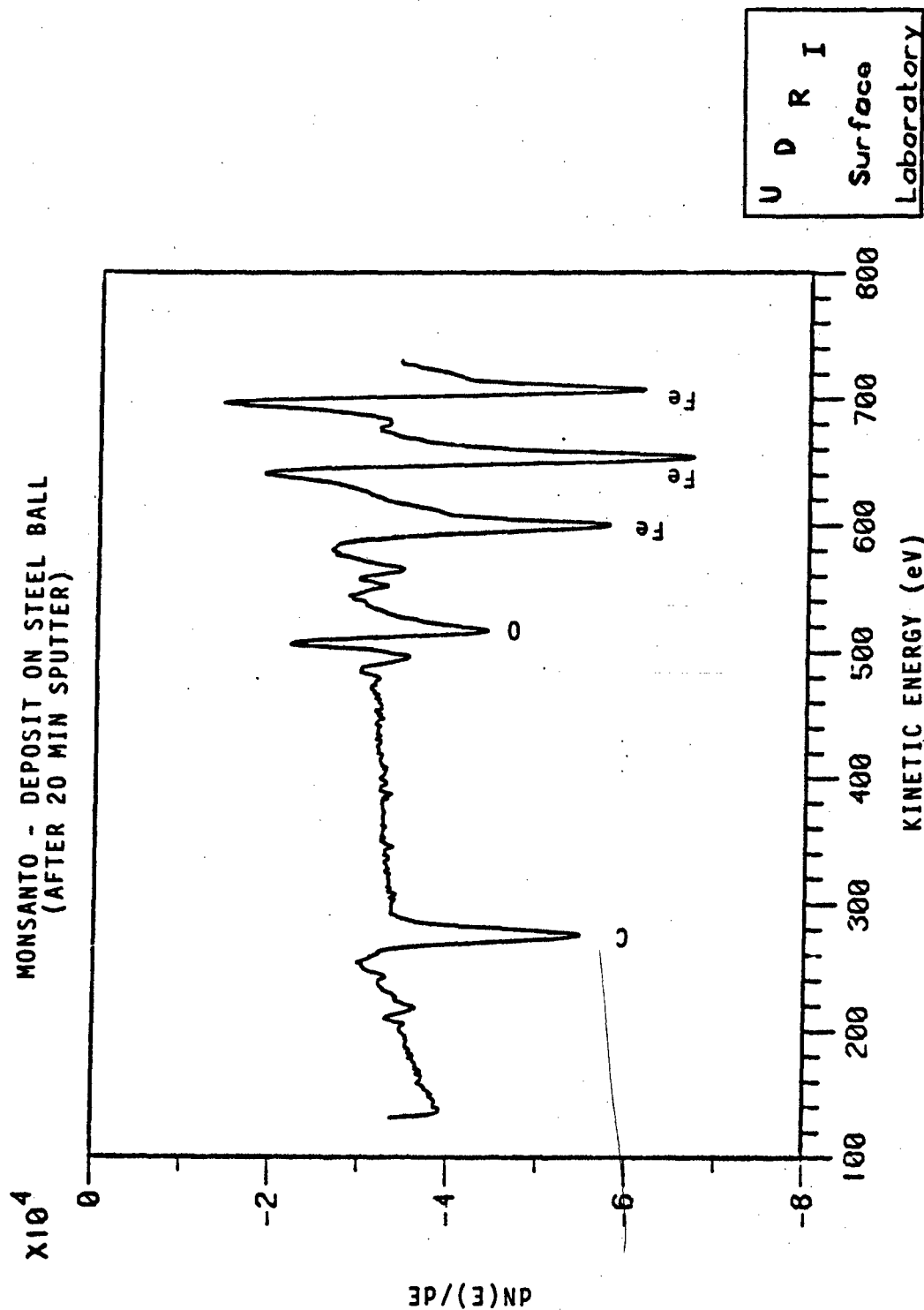
Figure 22. Analysis by AES, after 20 min sputter, of deposit on steel ball (83-POSF-1326).
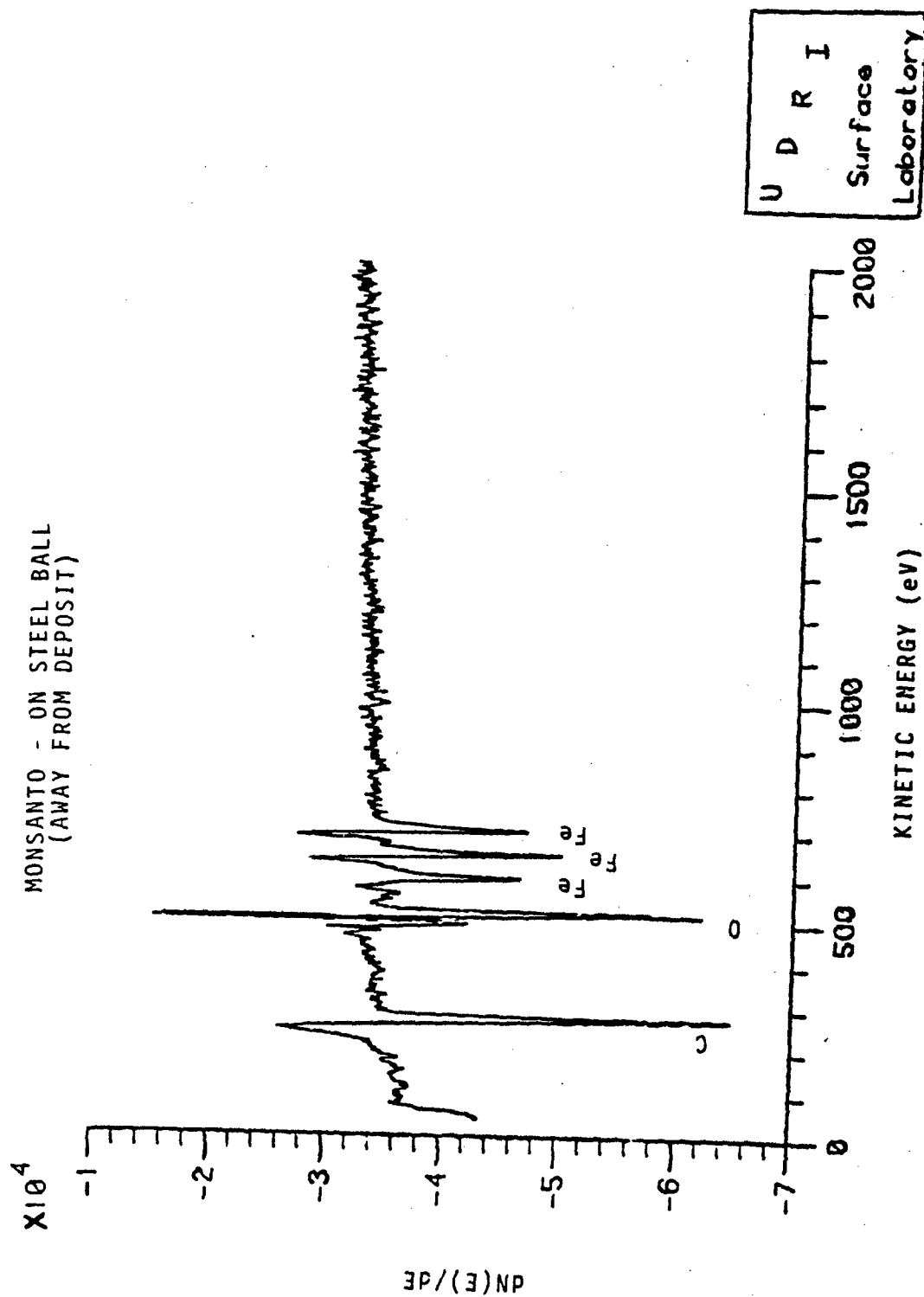
Figure 23. Analysis by AES on steel ball (83-POSF-1326) away from deposit.

MONSANTO - ON STEEL BALL AWAY FROM DEPOST
(AFTER 20 MIN SPUTTER)

KINETIC ENERGY (eV)

dN(E)/dE

X10⁵

U D R I
Surface
Laboratory

Figure 24. Analysis by AES on steel ball (83-POSF-1325)
away from deposit after 20 min sputter.

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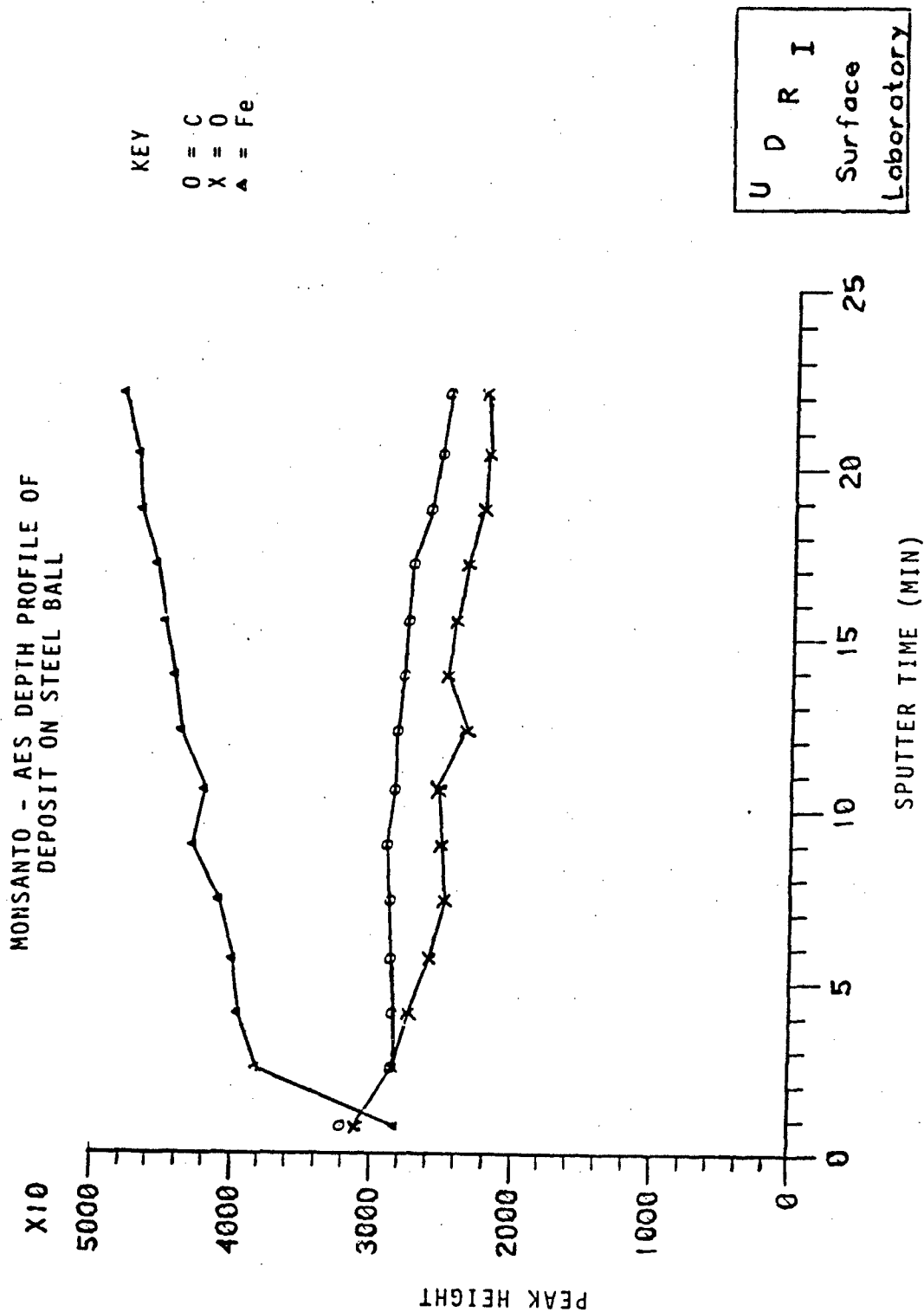
Figure 25. Depth profile by AES of deposit on steel ball (83-POSF-1326).

135
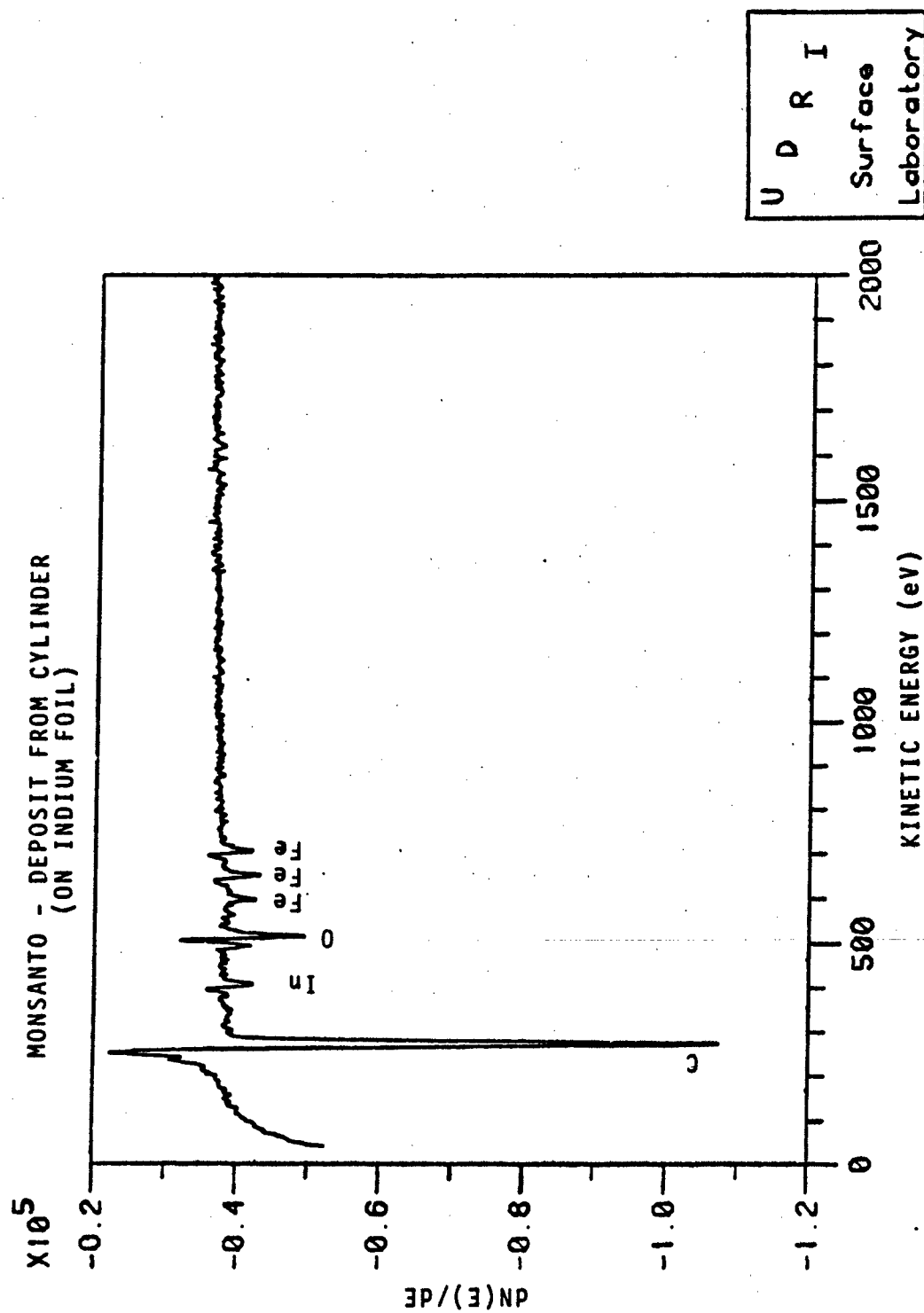
Figure 26. Analysis by AES on deposit from cylinder (83-POSF-1320).

JFTOT Samples

Problems had also been encountered with regard to the high temperature stability of some of the fuels as measured by the Jet Fuel Thermal Oxidation Tester (JFTOT). In order to determine possible fuel problems as contributing to JFTOT failures, a series of O-rings from this testing was examined by EDXA. Identification of samples follows.

(1) O-ring from JFTOT #6017 JP-4 filter assembly (83-POSF-1290)

(2) Unused O-ring from JFTOT tube assembly

(3) O-ring from around JFTOT tube assembly before heater on

(4) Blank specimen holder (with 1/7 mil Mylar film)

EDXA spectra showed no great differences. It can be noted that barium was observed only in the O-ring from JFTOT Run #6017 filter assembly, and that chlorine was seen only in the O-ring from around the tube assembly before the heater was turned on.

Conclusion

From the extensive testing that was carried out on this task, one fuel (83-POSF-1312) was found to be different from the other fuels. Fuel -1312 contained detectable levels of copper, boron, barium, chromium, lead, and silicon while the other fuels tested showed none. The sodium level of -1312 was also several times higher than the other fuels tested. Finally, the particulate level in -1312 was higher than any other fuel tested. It would seem reasonable that these fuel properties, which would not have been revealed by the specification tests in MIL-T-5624L, could have contributed to some of the problems encountered.

5. CHROMATOGRAPHIC SEPARATION OF FOUR SHALE-DERIVED FUELS

Four shale-derived fuels (sample numbers 81-POSF-005, 82-POSF-0511, 82-POSF-0321, and 83-POSF-0986) were submitted for column chromatographic separations. A silica gel column was used to strip polar components from these samples. Most of the polar materials were eluted with chloroform; those remaining were flushed off with ethanol. Samples of both the chloroform and ethanol fractions from each of the four fuels were submitted to Virginia Polytechnic Institute and to the Aero Propulsion Laboratory for further evaluation.

Analyses of the fuel fractions were also carried out by Monsanto using a Hewlett-Packard 5840 GC equipped with a nitrogen-phosphorus detector (NPD). In this way, information was gathered concerning the nitrogen content of the fuels, as well as to point out differences in the polar materials eluted by the two solvents.

The performance of the NPD was checked using a standard containing six different nitrogen compounds. These compounds were: (a) pyridine, (b) 2-vinylpyridine, (c) aniline, (d) nitrobenzene, (e) 1-naphthylamine, and (f) diphenylamine. The response of the NPD to these compounds at the 10 ppm level in hexane is shown in Figure 27. (Pyridine was not seen--possibly being obscured by the solvent peak). In looking at the chromatographically--separated fraction, no attempt was made to achieve quantitation, but merely to get an idea of the number and boiling range of nitrogen compounds in the eluates.

One sample, 82-POSF-0321, showed a wide range of nitrogenous compounds of polarity differences such that both eluants, chloroform and ethanol, stripped appreciable amounts from the fuel. Results of GC work done with the NPD are shown in Figures 28 and 29. The chloroform eluate (Figure 28) has a multitude of peaks falling in the volatility range of $C_{13}-C_{17}$ hydrocarbons, while the ethanol
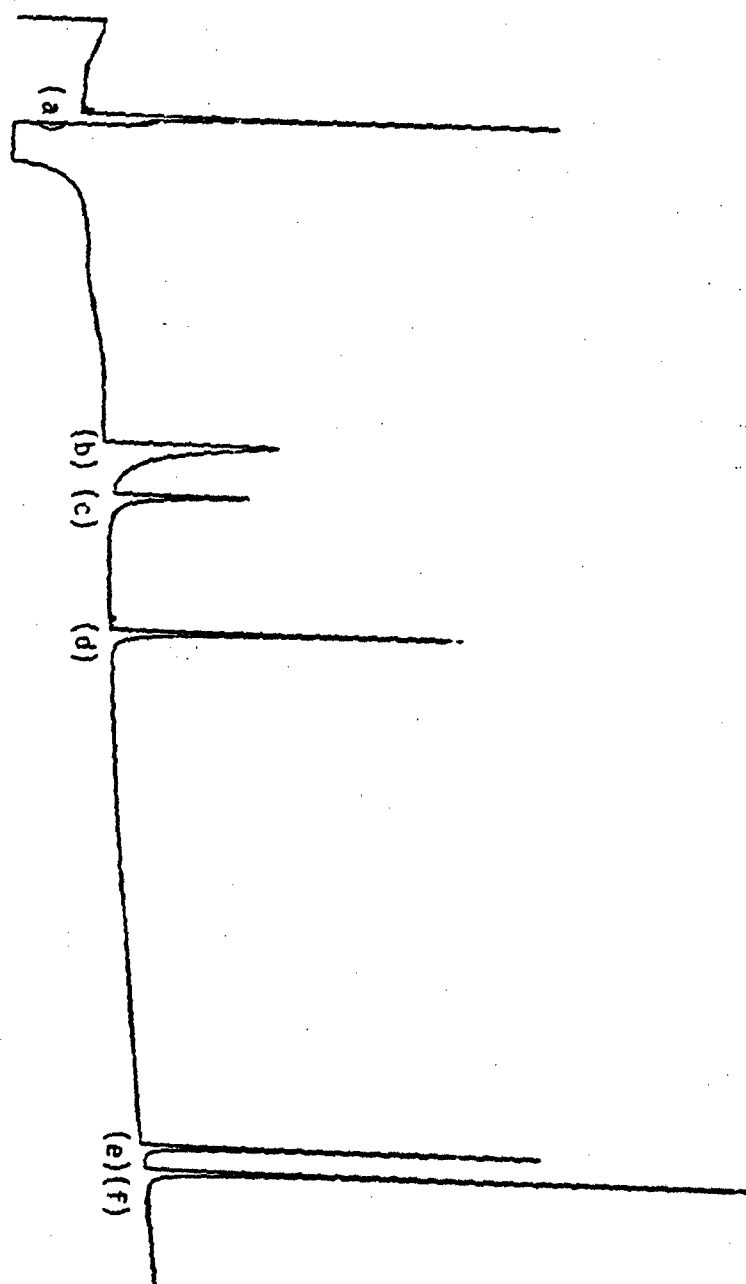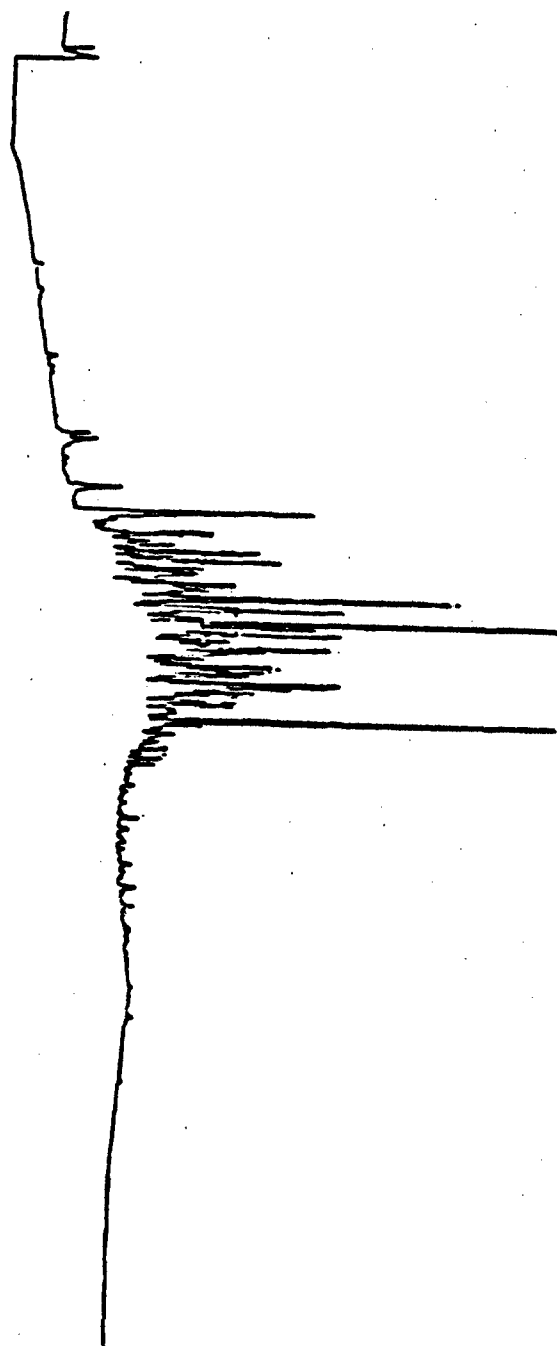
Figure 27. Nitrogen compounds standard.

Figure 28. Chloroform eluate of sample 83-POSF-0321.

eluate (Figure 29) is lighter with many peaks in the volatility range of C_{10}-C_{14} hydrocarbons. In Figure 30 is displayed the GC trace for the chloroform eluate of sample 82-POSF-0511. This fraction is similar to that from sample -0321 which was discussed previously. However, in contrast, the ethanol fraction of -0511 showed practically no nitrogen compounds (is not displayed).

Fractions of sample 81-POSF-005 were markedly different from the previous two samples discussed. Figure 31 is the chloroform eluate, and indicates only very slight amounts of nitrogenous material (large peak at ~2 minutes is internal standard diphenylamine). Conversely, in Figure 32 the ethanol eluate shows a fairly substantial wide boiling range of nitrogen compounds. The fourth sample, 83-POSF-0986, apparently contained a low level of nitrogen compounds, as the chloroform eluate, which is not displayed, showed practically no nitrogen compounds.
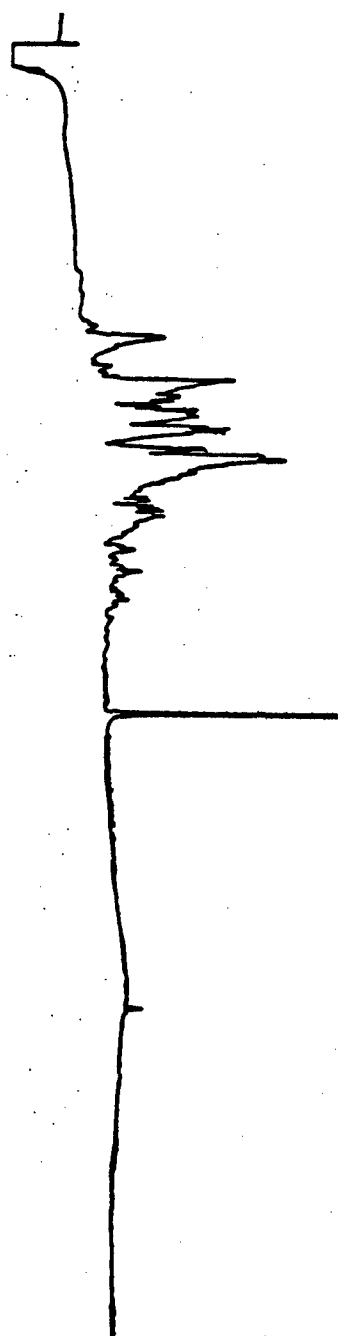
Figure 29.   Ethanol eluate of sample 82-POSF-0321.

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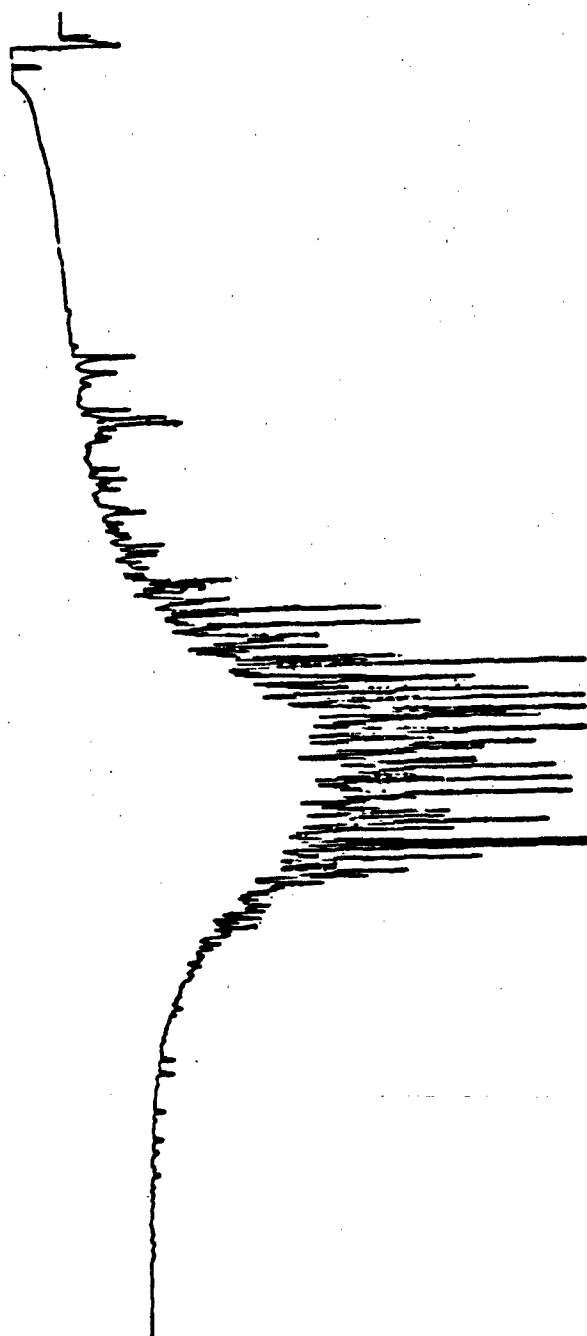
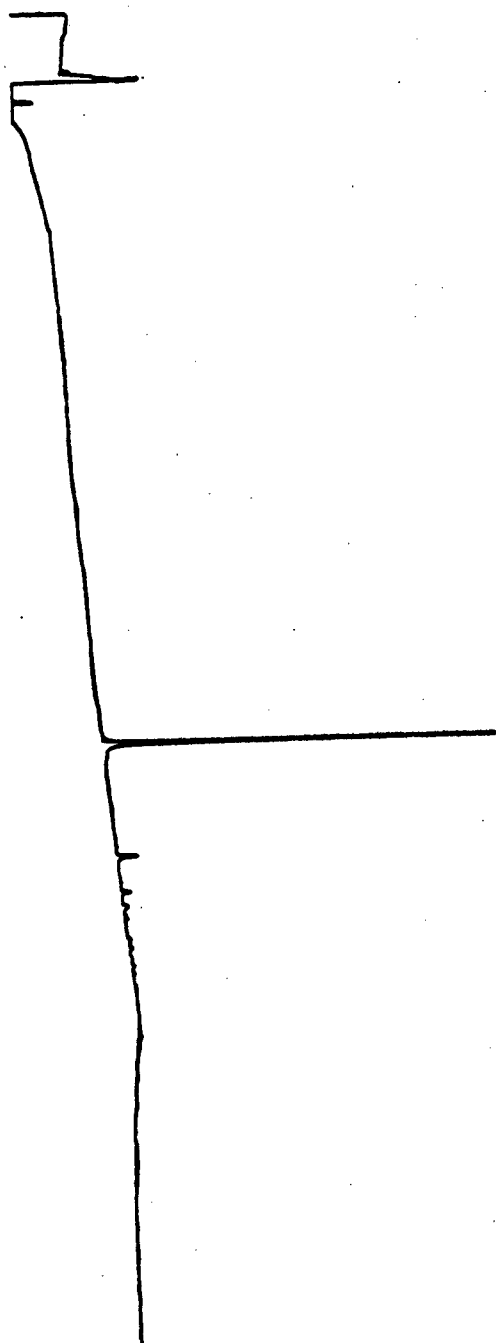Figure 30.   Chloroform eluate of sample 82-POSF-0511.

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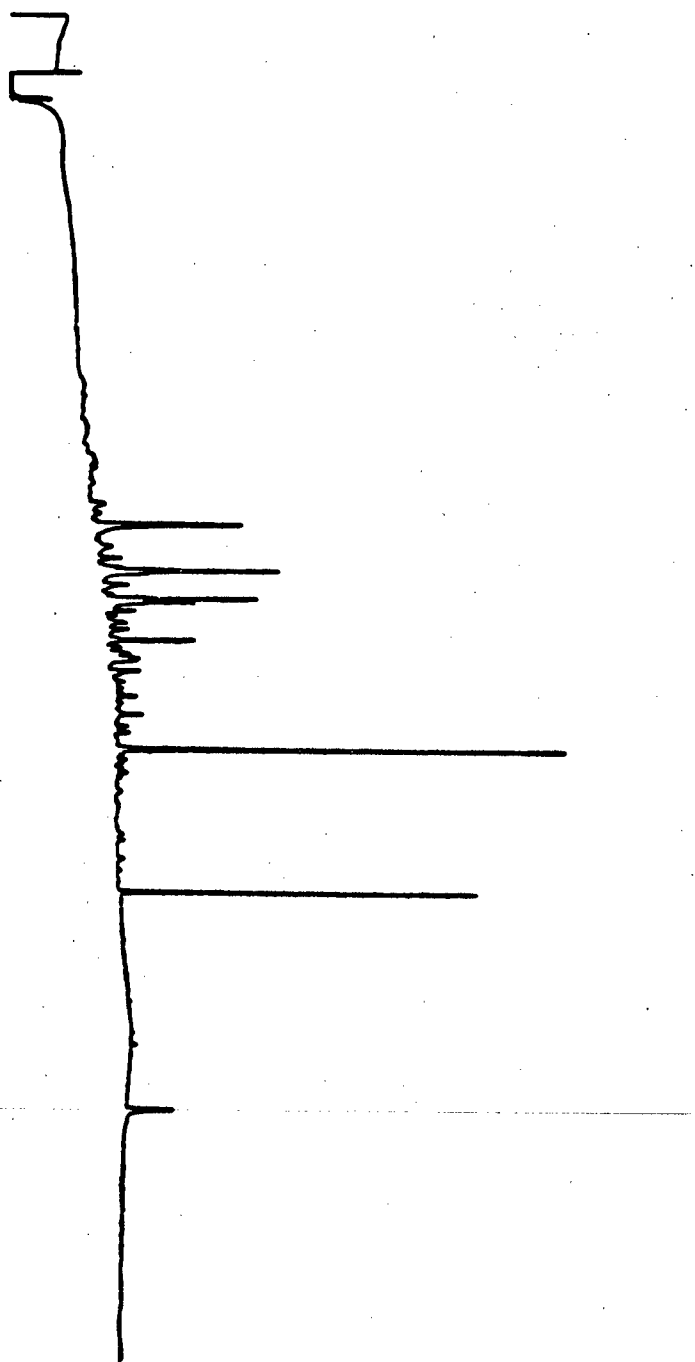Figure 31. Chloroform eluate of sample 81-POSF-005.

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Figure 32. Ethanol eluate of sample 81-POSF-005.

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APPENDIX A

SPECIFIC TEST METHODS FOR FUEL
CHARACTERIZATIONS DESCRIBED IN THIS REPORT

Several physical and chemical property tests were repeatedly conducted for a number of the projects described in this report. For the sake of conciseness and to avoid repetition, the test methods are described in this Appendix only.

DENSITY AND SPECIFIC GRAVITY

This method covers the laboratory determination, using a pyrex dilatometer, of the density of fuels normally handled as liquids. The dilatometer method is most suitable for determining the density of mobile transparent liquids ranging from 100°F to -65°F. In this procedure, the liquid is introduced into a clean, weighed dilatometer which is then reweighed. The sample is brought to the prescribed temperature by immersing the dilatometer in a vertical position into a constant temperature bath. After temperature equilibrium has been reached, the dilatometer scale is read by means of a cathetometer.

Density is defined as the mass of liquid per unit volume at prescribed temperature. In this method, the unit of mass is the gram and the unit of volume, the milliliter.

The dilatomer should be thoroughly cleaned with chromic acid cleaning solution or laboratory detergent, then rinsed well with distilled water and dried at 105° to 110°C, or rinsed with pure dry acetone and dried by applying an aspirator to the open end of the dilatometer. Cleaning should be performed in this manner in order to have a sharply defined miniscus during calibration of the dilatometer. Ordinarily, the dilatometer may be cleaned between test determinations by washing with a suitable solvent, such

as toluene, and rinsing with pure, dry acetone. Periodic cleaning with glassware detergent solution is recommended.

The volume of the dilatometer, when equilibrated at various test temperatures must be determined by means of a cathetometer. This is called the K value for that temperature. Freshly-boiled and cooled distilled water can be used for calibrating at 70°F and 100°F and good reagent grade solvents can be used for determining the K values at lower temperatures. Densities of the solvents at low temperature can be determined by using the International Critical Tables and the density of water at various temperatures can be found in the Handbook of Chemistry and Physics. When using the cathetometer, measure the span between two graduation marks on the dilatometer. This will be approximately 0.190 cm for a graduation interval of 0.05 ml. This should be measured at several places to assure consistency. Having determined the span of a 0.05 ml interval, the volume of the liquid can then be interpolated. It is more precise to determine the volume by the cathetometer than to estimate it by visual observation.

<u>Procedure</u> - Adjust a constant temperature bath to maintain the prescribed temperature. Weigh the clean, dry dilatometer and stopper to the nearest 0.1 mg and record the weight.

Fill the dilatometer to approximately the 1.5 ml graduation mark on the dilatometer by means of a hypodermic syringe. Remove any bubbles that might have been formed while transferring the sample.

Weigh the stoppered dilatometer and sample to the nearest 0.1 mg. Record the weight. Place the dilatometer in a suitable holder in the constant temperature bath adjusted to the test temperature within ±0.05°F. When the sample has reached equilibrium (about 15 minutes) take readings of the miniscus by means of a cathetometer. Read the cathetometer to the nearest 0.005 cm. Take several readings of the miniscus until reproducible readings

are obtained. After consistent readings have been made, remove
the dilatometer from the bath and clean with a suitable solvent,
rinse with pure dry acetone and proceed to the next test.

Calculations

$$\text{density, g/cc} = \frac{m}{K-R} \tag{1}$$

where m = mass of sample in grams

    K = dilatometer constant at given temperature,
        volume of dilatometer to full mark

    R = dilatometer reading at given temperature,
        ml below full mark

TRUE VAPOR PRESSURE

True vapor pressure is the maximum vapor pressure that a volatile
mixture such as an aircraft fuel can exert at a given temperature.
In theory, this property should be measured in the absence of sam-
ple vapor because vaporization of a portion of the sample changes
the composition of the mixture and thus changes the vapor pressure.
In practice, true vapor pressure must be measured at such a small
vapor-to-liquid ratio that any change in the composition of the
fluid produces a negligible change in vapor pressure, i.e., within
the experimental error of the method.

For true vapor pressure measurements as a function of temperature,
a micro vapor pressure apparatus, as described in ASTM D 2551-80
was used. The micro vapor pressure apparatus incorporates a
mercury-sealed orifice for sample introduction, and the entire
unit is surrounded by a glass outer jacket through which fluid
from a constant temperature bath is circulated. Using this
device, a known volume of sample is introduced into an evacuated,
temperature-controlled chamber of known volume. The rise in pres-
sure in the bulb is due to the vapor pressure of the sample and

the partial pressure of any dissolved air. In order to obtain a
vapor pressure measurement without a contribution due to air, the
air was first removed from the sample before the measurement was
conducted. A cryogenic degassing procedure is used for this
purpose.

Classically the vapor pressure temperature relationship is
expressed by:

$$\log P = A - B/T \tag{2}$$

where A and B are constants, P = absolute pressure and T =
absolute temperature. The line resulting from a plot of this
equation on semilog paper is useful over a limited range for
estimating vapor pressure at temperatures intermediate to those
at which measurements were taken.

HYDROCARBON TYPES BY MASS SPECTROMETRY

Hydrocarbon-type analyses were conducted by three separate mass
spectral methods, depending on the fuel. A modification of ASTM
Method D 2789 was used for JP-4 and gasoline-type fuels. ASTM
Method D 2425, which first requires an ASTM D 2549 separation of
the fuel into aromatic and paraffinic fractions, was utilized
mainly for diesel fuels. Monsanto Method 21-PQ-38-63, developed
for hydrocarbon feed stocks with an average carbon number in the
range of 12 to 13, was used for JP-8 type fuels. Nonstandard
fuels were usually analyzed by more than one mass spectral method.
All of these analyses are based on the summation of characteristic
mass spectral lines for each compound type. A matrix of n equa-
tions, relating each of n hydrocarbon types to the summed peak
values, is constructed. A computer solution of these simultaneous
equations provides a quantitative measure of each compound type
present.

As noted above, ASTM D 2425 analysis must be preceded by a separation of fuel aromatics from nonaromatics using a procedure such as that described in ASTM D 2429. The D 2549 method, as currently presented in Part 24 of the 1980 Annual Book of Standards, required a small procedural modification in order to be used for JP-8. This modification did not change the essential features of the separation, but only involved the method for removing the chromatographic solvent. The modified methodology was developed in MRC laboratories and has been employed for a number of years. An official modification of ASTM D 2549 to achieve the same effect is under study by ASTM Committee D-2 on Petroleum Products and Lubricants. By the ASTM D 2549 procedure, a steam bath is employed to evaporate solvent from the fractions obtained by elution chromatography. In the MRC modification, no heat is applied. Instead, a stream of dry nitrogen is used for desolvation. Evaporation of solvent, in fact, reduces the temperature to below ambient. After the major part of the solvent has evaporated, the weight of the fraction is carefully monitored as the final traces of solvent are removed. Complete removal of solvent is signalled by a marked decrease in the slope of the time/weight loss curve, or in some cases, by the attainment of a constant weight. MRC analysts are experienced in this procedure, which requires a short period of dedicated attention for the processing of each fraction by the analyst.